# DEVELOPMENT OF EQUIPMENT AND PROCESS INFORMATION RELATED TO THE FLASH HYDROPYROLYSIS OF COAL

FINAL REPORT ON

TASK 2 - COMMISSIONING AND SHAKEDOWN

BY

KILBORN LIMITED

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## 1.0 SUMMARY

This report describes the results of the test work performed during commissioning and shakedown of the flash hydropyrolysis (FHP) unit. The test work can be divided into three categories:

- demonstration of the function of coal feeder
- pressure testing of the FHP unit
- pilot plant runs.

All of the tests were carried out prior to and on December 23, 1983. The operating conditions for these tests were in accordance with the specifications given in the DSS Contract 18SQ.23440-9-9151 Amendment No. 2. The test results fulfilled the requirements of the same specifications.

Following successful completion of Task 2 - Commissioning and Shakedown, the FHP unit was delivered on December 23, 1983:

F.O.B. Ontario Research Foundation
Sheridan Park Research Community
Pilot Plant Area
Mississauga, Ontario.

## 2.0 INTRODUCTION

In June 1980, Kilborn Limited of Toronto was awarded a contract by Supply and Services Canada, on behalf of CANMET, to design, construct and operate a bench-scale, high-pressure and temperature flash hydropyrolysis (FHP) unit. The purpose of this unit is to develop fundamental information for the processing of Canadian coals using FHP technology.

The original CANMET FHP contract consisted of four tasks, namely:

Task 1. Design and construction of the FHP Unit.
Task 2. Commissioning and Shakedown of the FHP Unit.
Task 3. Study of the Effect of Process Variables on Product Composition and Yield
Task 4. Recommissioning of the FHP Unit at the Energy Research Laboratory.

Kilborn, as prime contractor, was responsible for all aspects of the CANMET FHP contract, including design, construction, initial testing and commissioning of the continuous bench-scale facility. ORF, as subcontractor, was responsible for the day-to-day operation of the FHP Unit and analysis of the product samples. CANMET, as Scientific Authority, was responsible for the scientific and technological content of the contract.

In May 1982, DSS/CANMET issued Contract Amendment No. 1 which deleted the entire Tasks 3 and 4 from the contract work. Task 2 was revised to include pressure testing of the FHP unit, coal feeder demonstration, and 5 (five) successful test runs with at least 1 (one) run under maximum operating conditions of 1000$^{o}$C and 17.25 MPa.

Task 1 was completed on September 30, 1982. The FHP Unit was constructed in the Pilot Plant Area of Ontario Research Foundation, Mississauga, Ontario.

Initial test work for Task 2 - Commissioning and Shakedown of the FHP Unit started in October 1982. During the period from October 1982 to the end of February 1983, a number of tests and related activities were carried out, including:

- Coal feeder tests and modification of the coal feeder system, e.g. refabricating augers, acquisition of a complete series of auger motors, modifying coal hopper, etc.

- Pressure testing of the FHP unit and correction of mechanical defects, e.g. replacing/repairing pneumatically-operated valves, various gaskets, rupture discs, check valves and others, re-machining of char trap lower flange surface, etc.

- Five pilot plant runs, which were unsuccessful because of the failure of the hydrogen mass flow meter and the inadequacy of the hydrogen preheater. The flow meter was returned to the manufacturer (Tylan) for repair and was received on March 10, 1983, too late to be used in any of these pilot plant runs.

Discussions with DSS and CANMET led to Contract Amendment No. 2, issued on May 30, 1983. Based on strength limitations of the preheater and reactor pipe, the required maximum operating conditions of test runs were lowered (see Appendix A). A complete redesign and replacement of the hydrogen preheater system took place during the period of July-October 1983. The final work on commissioning and shakedown of the FHP unit commenced on October 17, 1983 and all of the test activities were completed on schedule by December 23, 1983. The sequence of events was as follows:

- The coal feeder demonstration tests and the 5-hour pressure testing of the FHP unit with 1,500 psi cold helium were successfully completed before the end of October 1983.

- The successful pilot plant runs and final pressure testing of the unit were completed during the period of November 28 to December 23, 1983. The slow progress in November 1983 was due to another failure of the Tylan hydrogen mass flow meter on October 31, 1983 and the failure of the reactor thermocouple fillet weld on November 21, 1983.

# 3.0 DEMONSTRATION OF FUNCTION OF THE COAL FEEDER

## 3.1 Test Requirements

According to the DSS contract document referred to in Section 1.0, the requirements of the coal feeder tests are:

- It shall be demonstrated that the coal feeder is capable of feeding controllable amounts of coal without blockage problems. The range of coal feed rate shall be between 100 and 1000 g/h. Calibration between the speed of the auger motor and coal feed rate shall be established for 100 mesh particle size, Alberta Forestburg sub-bituminous coal.

## 3.2 Coal Feeder System

The key elements of the present coal feeder system are:

- a cone-bottom hopper capable of holding 1,400 g of Alberta Forestburg sub-bituminous "C" coal - the feed coal used in all of the coal feeder tests and pilot plant runs.

- 5 Emerson motors of 6, 8, 12, 20 and 25 rpm

- an auger of 6 turns per inch (auger A) and an auger of 4 turns per inch (auger B).

## 3.3 Calibration Test Program

Prior to the coal feeder demonstration tests and pilot plant runs, the feed coal was ground and sized to 25% -48 to +100 mesh, 75% -100 to +150 mesh. With the selective combination of the Emerson motors and augers, a series of coal feeder calibration tests were carried out during the week of October 17, 1983. In all, 17 tests were done, including 10 bench tests and 7 tests performed while the feeder system was mounted in place on top of the reactor. Each test lasted 60 minutes. The results of the tests are given in Tables 1 to 3.

Table 1 indicates the measured motor speed (rpm) in comparison with the nominal motor speed. It appears that the actual speed of the motors differs from the nominal speed, but this variation does not follow any particular pattern. It is for this reason that the actual speed of the motors, rather than the nominal speed, is used in determining the correlation between the coal feed rate in grams per hour versus the motor speed in rpm (Figure 1).

## 3.4 Bench Test Results

For the bench coal feeder tests, 10 combinations of motors and augers were obtained. The measured 1-hour coal feed rates (in grams) are given in Table 2. The coal feed rate varies from 246 to 916 g/h for Auger A and from 364 to 1,345 g/h for Auger B.

## 3.5 In-Place Test Results

For the in-place coal feeder tests, 7 combinations of motors and augers were obtained: 20 rpm motor with Auger A; 6, 8 and 12 rpm motors with Augers A & B. The measured 1 hour coal feed rates (in grams) are given in Table 3. The coal feed rate varies from 218 to 1,010 g/h for Auger A and from 322 to 693 g/h for Auger B.

## 3.6 Observations and Conclusions

Results of both the bench tests and the in-place tests are shown in Figure 1, in which the measured coal feed rates in g/h are plotted against the actual motor speeds in rpm. Following are the observations of the test results:

a. The in-place coal feeder with Auger A has a coal feed capacity of 1,010 g/h.

b. For both bench and in-place coal feeder tests Auger B provides a greater feed capacity than Auger A, i.e. while using the same motor the coal feed rate with longer pitch Auger B is always higher than that with the short-pitch Auger A.

c. It appears that the coal feeder results are not reliable above a nominal motor speed of 20 rpm. Proper feedrate can be obtained below 20 rpm with the appropriate auger.

d. It also appears that, while using the same motor and auger at lower motor speeds (6, 8, 12 rpm), the in-place coal feeder gives a slightly lower feed rate compared to the bench coal feeder test data. However, with 20 rpm motor, the in-place coal feeder has a higher feed rate than the bench coal feeder.

The above results therefore demonstrate that:

- The capability of the coal feeder system fulfills the test requirements described in Section 3.1.

- The coal feeder system is able to support a 90 minute pilot plant run at a coal feed rate of 930 g/h.

- The coal feed rate can be increased beyond 1,000 g/h with the combination of the 20 rpm motor and Auger B.

TABLE 1. ACTUAL SPEED (RPM) OF COAL FEEDER MOTORS

| NOMINAL MOTOR SPEED, rpm | ACTUAL MOTOR SPEED, rpm |
|---|---|
| 25 | 22.5 |
| 20 | 20.75 |
| 12 | 9.25 |
| 8 | 8.75 |
| 6 | 8.0 |

TABLE 2. RESULTS OF THE BENCH COAL FEEDER DEMONSTRATION TESTS

Feed Coal: Alberta Forestburg sub-bituminous "C" coal, ground and sized to 25% -48 to +100 mesh, 75% -100 to +150 mesh.

Duration of Each Test: 60 minutes.

| TEST NO. | MOTOR (Nominal rpm) | AUGER* | COAL FEED RATE (g/h) |
|---|---|---|---|
| 1 | 25 | A | 850 |
| 2 | 25 | B | 1,320 |
| 3 | 20 | A | 916 |
| 4 | 20 | B | 1,345 |
| 5 | 12 | A | 427 |
| 6 | 12 | B | 706 |
| 7 | 8 | A | 360 |
| 8 | 8 | B | 632 |
| 9 | 6 | A | 246 |
| 10 | 6 | B | 364 |

*Auger A: 6 turns per inch
Auger B: 4 turns per inch

TABLE 3. RESULTS OF THE IN-PLACE COAL FEEDER DEMONSTRATION TESTS

Feed Coal: Alberta Forestburg sub-bituminous "C" coal, ground and sized to 25% -48 to +100 mesh, 75% -100 to +150 mesh.

Duration of Each Test: 60 minutes.

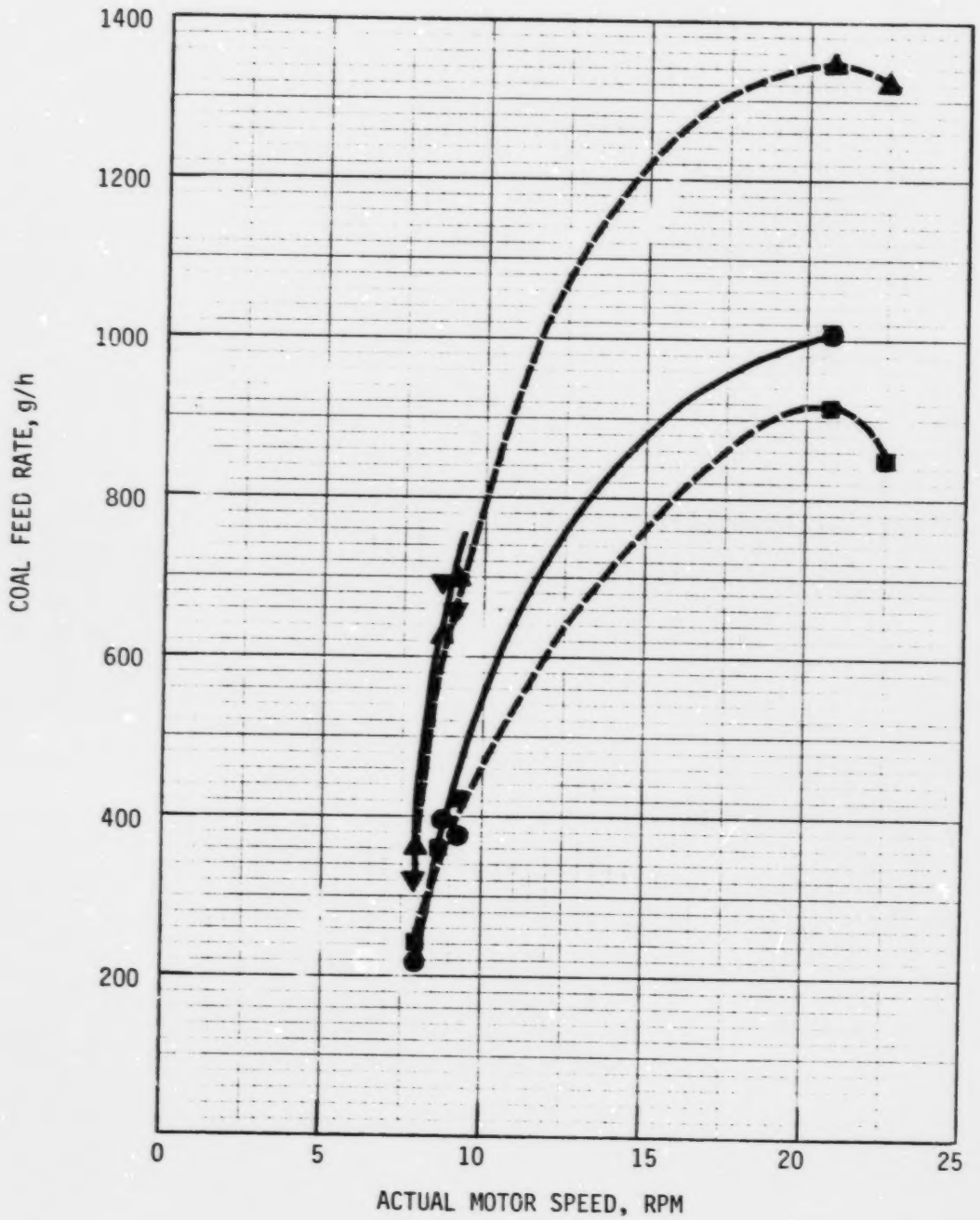
| TEST NO. | MOTOR (Nominal rpm) | AUGER* | COAL FEED RATE (g/h) |
|---|---|---|---|
| 1 | 20 | A | 1,010 |
| 2 | 12 | A | 380.5 |
| 3 | 12 | B | 662 |
| 4 | 8 | A | 396 |
| 5 | 8 | B | 693 |
| 6 | 6 | A | 218 |
| 7 | 6 | B | 322 |

*Auger A: 6 turns per inch
Auger B: 4 turns per inch

FIGURE 1. COAL FEEDER CALIBRATION TEST RESULTS

| Motor rpm | Actual rpm |
|---|---|
| 25 | 22.5 |
| 20 | 20.75 |
| 12 | 9.25 |
| 8 | 8.75 |
| 6 | 8.0 |

■ Auger A, Bench Test
▲ Auger B, Bench Test
● Auger A, In-Place Test
▼ Auger B, In-Place Test



# 4.0 PRESSURE TESTING OF THE FHP UNIT

## 4.1 Test Requirements

The contract documents specify that:

- This involves demonstrating that the unit can hold pressure for a five hour period while the vessels are at elevated temperature. Thus with the reactor maintained at 800$^{o}$C at a pressure of 2,000 psi (13.8 MPa), the pressure shall not drop by more than 100 psi (0.69 MPa) in a five (5) hour period.

## 4.2 Summary of Pressure Test Programs

Initial work on pressure testing of the FHP unit with nitrogen and helium at room temperature was carried out in the period of December 1982 - January 1983. This resulted in detection and subsequent elimination of many leak sources. Towards the end of January 1983 the entire FHP unit was considered sealed tight.

On November 28, 1983, a successful pressure testing of the FHP unit with helium at 10.45 MPa (1500 psig) and room temperature was obtained. The pressure drop for a 5 hour period was 0.52 MPa (75 psi).

During the week of December 19, 1983, two final pressure tests were carried out with the reactor at 800$^{o}$C and 13.9 MPa (2000 psig). In the second of these pressure tests, the pressure drop for a 5 hour period was 0.21 MPa (30 psi).

## 4.3 Initial Pressure Tests

During the period of December 1982 to January 1983, initial pressure tests with nitrogen and helium at room temperature were carried out according to the following schedule:

| Initial System Pressure, psig | Pressure Increment, psig | Test Pressure, psig | Pressure Holding Duration, min. |
|---|---|---|---|
| 0 | 25 | 25 | 10 |
| 25 | 25 | 50 | 10 |
| 50 | 25 | 75 | 10 |
| 75 | 25 | 100 | 10 |
| 100 | 50 | 150 | 20 |
| 150 | 50 | 200 | 20 |
| 200 | 150 | 350 | 20 |
| 350 | 150 | 500 | 30 |
| 500 | 500 | 1000 | 30 |
| 1000 | 500 | 1500 | 300 |

During the holding period at each test pressure, leak tests were performed using either:

- a soap solution applied at flanged, screwed and welded joints, or
- an ultrasonic detector.

Corrective measures were taken to eliminate the leak sources, including:

- tightening the flange bolts and loose connections
- modification of thermocouple connections
- replacement of improper or defective parts, e.g. broken gaskets, inadequate rupture disc connections, leaking pneumatically-operated valves, plugged line filter, vibrating check valve, etc.
- re-machining the lower flange surface of the char trap, to offset the surface distortion.

With the repetitive pressure/leak tests and subsequent corrective mechanical work, the entire FHP unit was sealed satisfactorily tight towards the end of January 1983.

As the installation of the modified hydrogen preheater system was nearing completion in October 1983, the FHP unit was pressure tested again with nitrogen and helium at ambient temperature. Throughout this process, various leak sources were detected and remedial measures taken to eliminate the leaks, e.g. replacing gaskets, applying sealants and tightening connections.

On October 28, 1983, the FHP unit was pressurized to 10.45 MPa (1500 psig) with helium at room temperature and this pressure was held for a period of 5 hours. The total pressure drop for this period was 0.52 MPa (75 psi) which was considered satisfactory. The only leak sources were the thin fibre gaskets of two 39.4 cm (15½") O.D. flange gaskets on the coal feeder. These were replaced with suitable lead gaskets. After torquing the flange bolts to 225 ft-lb, these leaks were finally stopped.

## 4.4 Final Pressure Tests

The final pressure tests were carried out, one on December 20, 1983, and the other on December 23, 1983. The results are given in Tables 4 and 5.

For both tests the reactor was first heated and maintained at 800°C, then the FHP unit was pressurized to 10.45 MPa (1500 psig) with helium and further to 13.9 MPa (2000 psig) with hydrogen. This was done because of the limitation of available pressure in the helium cylinder.

Pressure Test No. 1 was aborted because the system pressure dropped 0.34 MPa (50 psi) within 41 minutes. The leak sources were found to be:

(a) the flange gasket of the water-cooled condenser product trap (this gasket was not replaced prior to the pressure test because of its unavailability), and

(b) the fitting of the pressure transmitter located in the outlet line of the char trap. Both of the above gaskets and fittings were replaced before Pressure Test No. 2.

For Pressure Test No. 2 the total pressure drop for a 5-hour period was 0.21 MPa (30 psi), according to the actual reading of the char trap outlet pressure gauge. Within 5 hours the temperature in the coal feeder gradually increased from 16°C to 23°C, while the gas temperature in the preheater tube increased from about 30°C to 36°C. The temperature between the heating section and the cooling section of the reactor tube maintained at 180°C throughout the pressure test period.

## 4.5 Observations and Conclusions

From the results of both of the initial and final pressure tests, the following observations/conclusions are made:

- The FHP unit fulfills the pressure testing requirements described in Section 4.1.

- To ensure the tightness of the FHP unit, the flange gaskets (stainless steel/asbestos type) in the char trap and two product traps must be replaced before each test run.

TABLE 4. RESULTS OF PRESSURE TEST NO. 1[a]

| TIME, h | REACTOR TEMPERATURE, °C ZONE 1 | ZONE 2 | ZONE 3 | ZONE 4 | SYSTEM PRESSURE[b] psig (MPa) | COAL FEEDER TEMP., °C | REMARKS |
|---|---|---|---|---|---|---|---|
| 0:00[c] | 802 | 801 | 801 | 799 | 1,500 (10.45) | | System pressurized with helium. |
| 0:05 | 799 | 802 | 801 | 801 | 1,800 (12.51) | | Further pressurized with hydrogen. |
| 0:11 | 802 | 802 | 802 | 801 | 1,925 (13.37) | | |
| 0:18 | 802 | 802 | 802 | 801 | 2,015 (13.99) | | |
| 0:25 | 802 | 802 | 802 | 801 | 2,000 (13.89) | 21 | Pressure testing started. |
| 0:40 | 802 | 802 | 802 | 801 | 1,975 (13.72) | 21 | |
| 0:55 | 802 | 802 | 802 | 801 | 1,955 (13.58) | 21 | |
| 1.05[d] | 802 | 802 | 802 | 801 | 1,950 (13.54) | 21 | Pressure testing terminated. |

a. Prior to this pressure test, the flange gasket in the char trap was replaced. Flange gaskets in the water-cooled condenser and liquid nitrogen chiller product traps were not replaced because of the unavailability of new gaskets.

b. "System pressure" indicates the char trap outlet pressure reading on the central control panel.

c. At time 0:00 h, the temperatures of the heaters for Zones 1, 2, 3 & 4 of the reactor were set at 793, 800, 793 & 793°C, respectively.

d. At time 1:06 h, the system pressure dropped 0.34 MPa (50 psi) within 41 minutes. The pressure testing was terminated immediately. The leak sources were found to be the gasket in the water-cooled condenser product trap and the fitting of the pressure transmitter PT-1, located after the 5u filter in the outlet line of the char trap. This fitting was replaced.

TABLE 5. RESULTS OF PRESSURE TEST NO. 2[a]

| TIME, h | REACTOR TEMPERATURE, °C ZONE 1 | ZONE 2 | ZONE 3 | ZONE 4 | SYSTEM PRESSURE[b], psig (MPa) | COAL FEEDER TEMP., °C | PREHEATER[c] GAS TEMP., °C | REACTOR EXIT[d] TEMP., °C |
|---|---|---|---|---|---|---|---|---|
| 0:00[e] | 800 | 802 | 809 | 808 | 2,000 (13.89) | 16 | | |
| 0:30 | 801 | 801 | 800 | 800 | 2,000 (13.89) | 18 | | |
| 1:00 | 801 | 800 | 800 | 800 | 2,000 (13.89) | 19 | 32 | 180 |
| 1:30 | 800 | 800 | 800 | 800 | 1,990 (13.82) | 19 | 34 | 180 |
| 2:00 | 801 | 800 | 801 | 800 | 1,990 (13.82) | 20 | 34 | 180 |
| 2:30 | 801 | 801 | 801 | 801 | 1,985 (13.79) | 20 | 36 | 180 |
| 3:00 | 801 | 801 | 801 | 801 | 1,980 (13.79) | 21 | 36 | 180 |
| 3:30 | 801 | 801 | 801 | 801 | 1,975 (13.72) | 21 | 36 | 180 |
| 4:00 | 801 | 801 | 801 | 801 | 1,970 (13.68) | 22 | 36 | 180 |
| 4:30 | 801 | 801 | 801 | 801 | 1,965 (13.65) | 22 | 36 | 180 |
| 5:00 | 801 | 801 | 801 | 801 | 1,960 (13.61)[f] | 23 | 36 | 180 |

a. Prior to this pressure test, all of the flange gaskets in the char and product traps were replaced.

b. "System pressure" indicates the char trap outlet pressure reading on the central control panel.

c. "Preheater gas temperature" indicates the measured in-tube preheater temperature during the static pressure testing period.

d. "Reactor exit temperature" indicates the temperature measured at the outlet of the heating section of reactor.

e. At time 0:00 h, the temperatures of the heaters for Zones 1, 2, 3 & 4 of the reactor were set at 792, 798, 792 & 792°C, respectively; the pressure of the FHP unit was set at 13.89 MPa (2000 psig).

f. At the end of the 5-hour pressure testing, the actual reading of the char trap outlet pressure gauge was 13.68 MPa (1970 psig).

# 5.0 PILOT PLANT RUNS

## 5.1 Test Run Requirements

Contract Amendment No. 2 specifies that:

- Included in the shakedown operation will be at least five (5) pilot plant runs feeding a representative Canadian coal, specified by CANMET.
- Each run shall be targetted to last one (1) hour with two (2) runs, each being a minimum of thirty (30) minutes in duration, and three (3) runs each being a minimum of twenty (20) minutes in duration.
- Two (2) of the runs shall be at 700°C and 800°C respectively at pressure of 1,500 psi (10.3 MPa). The remaining three (3) runs shall be at temperatures and pressures of not less than 500°C and 1,000 psi (6.9 MPa). One run shall demonstrate a nominal hydrogen flow rate of 2 (two) kg per hour.
- The overall material balance on the system shall be demonstrated in a minimum of 5 (five) successful runs with various durations. The sum of the product weights, namely gases, entrapped liquids and chars shall be within (plus or minus) 15% of the weight of reactants (coal and hydrogen).

Detailed requirements on process condition controls, product samples and analyses are given in the Appendix A.

## 5.2 Summary of Test Run Programs

Based on the target process conditions set in Contract Amendment No. 1, five pilot plant runs were made between January 28 and February 25, 1983. These runs were performed in less than ideal conditions as a substitute flow control device had to be improvised to replace the hydrogen mass flow meter which failed during an initial pressure test prior to January 28, 1983. All of these tests were unsuccessful due to a variety of reasons such as inadequate heating capacity of

the hydrogen preheater system, rupture of the preheater pipe, insufficient hydrogen supply, hydrogen leakage from the flange gasket of the liquid-nitrogen chiller product trap and failure of the pressure reducer.

The above problems were corrected by:

a. Redesign of the hydrogen preheater system, utilizing a direct-resistance heating approach to replace the radiation-type furnace

b. Repairing and recalibrating the hydrogen mass flow meter by the manufacturer - Tylan Corporation in the U.S.A.

c. Installing two additional valve stations to the hydrogen header.

d. Replacing product trap gaskets for each test run.

e. Modification of the pressure reducing unit, utilizing a single pressure reducer to replace the two-step pressure reducers.

Among the five corrective measures, Items (b), (c), (d), (e) were carried out immediately, however, Item (b) was a time-consuming process. The repaired flow meter was only received after all of the initial trial runs were completed. In the meantime, a simple flow control device was improvised as a replacement and the effluent gas flow totalizer was used to measure the gas flow rate.

Item (a) was a major task requiring detailed engineering design and a long period (12-16 weeks) for delivery of the purchased equipment items and instrument. This was done after the issuing of the Contract Amendment No. 2 by the DSS. The installation of the modified hydrogen preheater system was completed on October 31, 1983.

During the initial pressure testing of the FHP unit on October 31, 1983, the hydrogen mass flow meter failed to function for the second time. It was returned to Tylan Corporation for repair and recalibration. The repaired flow meter was received by ORF on November 17, 1983.

With the hydrogen mass flow meter/controller and the hydrogen preheater system operational, an attempt was made on November 21, 1983, to perform a pilot plant run. However, during the start-up period, the fillet weld on the reactor exit thermocouple failed and the test run was aborted. This fillet weld was repaired and examined with dye penetrant.

Based on the test run requirements described in Section 5.1, a schedule of pilot plant runs was drawn up and six pilot plant runs were carried out during the period of November 28 - December 16, 1983. The process conditions were:

#6 34 min. - 500°C - 1,000 psig - 1.8 kg/h $H_2$ - 1.0 kg/h coal
#7 30 min. - 600°C - 1,000 psig - 1.0 kg/h $H_2$ - 1.0 kg/h coal
#8 31 min. - 700°C - 1,000 psig - 0.9 kg/h $H_2$ - 0.9 kg/h coal
#9 60 min. - 700°C - 1,500 psig - 0.9 kg/h $H_2$ - 0.9 kg/h coal
#10 46 min. - 800°C - 1,500 psig - 1.0 kg/h $H_2$ - 0.8 kg/h coal
#11 61 min. - 750°C - 1,000 psig - 1.0 kg/h $H_2$ - 0.7 kg/h coal

Among the above pilot plant runs, Run Nos. 6, 8, 9, 10 and 11 were successful but Run No. 7 failed in terms of material balance due to a hydrogen leak at the electrical isolater, which was located ahead of the hydrogen preheater.

## 5.3 Coal Preparation and Analysis

Prior to the coal feeder demonstration tests and pilot plant runs, approximately 34 kg (75 lb) of Alberta Forestburg sub-bituminous "C" coal was predried and pulverized according to the following procedure:

a. Spread the coal in flat trays and leave the trays in a dryer at 93°C (200°F) for 3 days.

b. Crush the predried coal in a roll crusher and use a screen to separate and collect the -100 to +150 mesh fraction.

c. Pulverize the oversize (+100 mesh) coal in a pulverizer. Use screens to separate and collect, separately, the two fractions, -48 to +100 mesh and -100 to +150 mesh. Discard the undersize (-150 mesh) coal.

d. Re-pulverize the oversize (+48 mesh) coal in the pulverizer. Repeat the screening and pulverizing processes until all oversize coal is pulverized.

The two collected fractions of coal were then mixed in a proportion of 25% -48 to +100 mesh and 75% -100 to +150 mesh. The total quantity of feed coal obtained in this manner was about 6.8 kg (15 lb) sufficient for at least six 1-hour pilot plant runs of 1 kg/h coal feed each.

The results of the feed coal analysis are given in Table 6.

## 5.4 Run Nos. 1-5

In the first two months of 1983 five pilot plant runs were attempted. During these runs unforeseen mechanical, equipment and instrument problems were encountered, including hydrogen flow meter failure, preheater pipe rupture, chiller product trap gasket leakage, pressure reducer failure, etc. As a result of these mechanical failures and equipment limitations, none of these runs produced satisfactory results. In Run No. 4 about 5 $dm^3$ of coal liquid was obtained, however, the run had to be terminated because of a minor fire resulting from a leakage of the gasket in the chiller product trap.

The results of the Run Nos. 1 to 5 are described briefly in this section. The records of these runs are given in Appendix B.

### Run No. 1

The Tylan hydrogen mass flow meter was not available for the first set of runs as it had failed during pressure testing of the FHP unit in January 1983 and had been returned to the manufacturer for repairs. In its place a simple flow control device was improvised.

However, this device did not indicate the flow rate. This shortfall was overcome by using the effluent gas flow totalizer readings and the pressure drop in the hydrogen cylinders to verify the actual gas flow rate.

In Run No. 1 the FHP unit was tested at various process conditions, i.e. different set temperatures, pressures and gas flow rates, in order to gain operating experience with the unit. The details are given in the Appendix B - Table 1.

In summary, the following observations were made:

a) The hydrogen preheater system was found to be inefficient. The pilot unit heat-up rate was very slow; furthermore, the reactor inlet (gas) temperature could not be raised beyond 700°C with the preheater furnace temperature set at 1100°C.

The hydrogen preheater system used in Run No. 1 consisted of:

- a 3.06 m (10 ft) long pipe made of ¼" Sch. 40 Inconel 601 piping, formed into an inverted "U" (see Appendix B - Figure 1), which was heated via radiation

- A Lindberg electric, horizontal furnace of 6.1 kW rated capacity.

b) The hydrogen supply/storage capacity was insufficient as a single 41.5 MPa (6000 psig) hydrogen cylinder could support a test run with 1 kg/h $H_2$ feed for a duration of 17 minutes; the compressor (1 scfm or 28.3 $dm^3$/min) running at full capacity during the test run would prolong the run by only 3 minutes. The lack of an adequate flow control device aggravated this hydrogen supply problem.

c) The coal feeder malfunctioned during the run as no residue was found in the char trap at the end of the run.

Immediate measures were taken to correct the above problems. These included:

a) Modification of the existing hydrogen preheater system and the reactor heaters. This was accomplished by increasing the voltage of the power supply to these heaters from 201 to 212V, thus increasing the maximum output of the hydrogen preheater and four reactor heaters to 6.8 kW and 1.6 kW each.

   - adding a loop to the existing Inconel 601 preheater pipe (see Appendix B - Figure 1), thus increasing the total preheater pipe length from 3.06 m (10 ft) to 4.68 m (15 ft) and the heat transfer area by 50%.

b) Modification of the hydrogen header to accommodate an additional two hydrogen cylinders.

c) Addition of cooling water coils around the lower flange of the coal feeder system to prevent overheating of the coal feeder motor.

Run No. 2

For Run No. 2 it was decided to use a "no-flow" approach to heat up the FHP unit, in order to shorten the heating-up period and to conserve helium gas. The startup plan included heat-up of the preheater and reactor heaters to set temperatures under "no-flow" conditions, introduction of helium flow and reestablishment of temperatures, introduction of hydrogen and introduction of coal feed as soon as temperatures with hydrogen flow stabilized.

The details of Run No. 2 are given in the Appendix B - Table 2.

It was observed that under "no flow" conditions, within 2.5 hours the preheater furnace reached 1164$^{o}$C and the four reactor heaters were at the set 1000$^{o}$C. Twenty minutes later the preheater furnace reached the set 1200$^{o}$C.

During the switchover from "no flow" conditions to helium flow conditions, the preheater pipe ruptured (see Appendix B - Figure 1). At the time of the rupture, the pressure in the FHP unit was 5.0 MPa (710 psig). It was obvious that the allowable stress of the preheater pipe was exceeded due to local overheating under "no flow" conditions.

Immediately after Run No. 2 the ruptured section of the preheater pipe was replaced and the welded connections x-rayed.

Run No. 3

For this run the furnace temperatures were set at lower levels than those of Run No. 2: 1050$^{o}$C for the preheater and 950$^{o}$C for the reactor. A similar "no flow" heatup procedure to that used in Run No. 2 was followed.

During the switchover from the helium to hydrogen gas flow, the preheater pipe ruptured again (see Appendix B - Figure 1). At the time of the rupture the system pressure was 10.4 MPa (1500 psig).

The details of Run No. 3 are given in the Appendix B - Table 3.

After the failure of Run No. 3, the following activities were carried out immediately:

a. Replacing the ruptured section of the preheater pipe, followed by x-ray examination of the welded connections.

b. Careful verification of maximum allowable operating conditions for the FHP unit, particularly the preheater pipe and the reactor tube.

c. Searching for more suitable Inconel material (higher allowable stress) to be used as preheater pipe.

Subsequent to a number of discussions with Inco personnel on the availability of Inconel materials and their rupture strengths, it was concluded that:

a) ¼" Sch. 40 (dn6) Inconel 601 piping was the best available material for use as the preheater pipe. Other more suitable materials that would allow higher operating temperature and pressure were long delivery and would require a large quantity order, therefore impractical.

b) The maximum allowable operating conditions for the present preheater pipe were to be: 870°C at 13.9 MPa (2000 psig).

In addition to above, the maximum allowable temperatures and pressures for the preheater pipe (¼" Sch. 40 Inconel 601 pipe) and the reactor tube (1¼" Sch. 80 Inconel 617 pipe) respectively under various operating conditions, based on a 100-hr rupture life, were calculated and presented in Figures 2 & 3. These figures are to be used to define the maximum process operating conditions.

Run No. 4

For Run No. 4 the temperatures of the preheater furnace and four reactor heaters were set at 850°C. The system pressure was set at 13.9 MPa (2000 psig).

The details of this run are given in the Appendix B - Table 4.

In this run, desired operating conditions were achieved with the system under hydrogen flow. A minor fire occurred after the coal feeder had been operational for about 4 minutes, being caused by leakage from the gasket of the chiller product trap. About 5 $dm^3$ of coal liquid was obtained from the water-cooled product trap.

Immediately after the run, the failed gasket was replaced with a thinner gasket of the same material and the flange bolts were tightened to specified torque limits.

Run No. 5

For this run the temperatures of the preheater furnace and four reactor heaters were set at 600°C. The planned system pressure was 10.4 MPa (1500 psig).

Operation of the FHP unit was relatively smooth with helium flow. As soon as the gas flow was switched over to hydrogen feed, no flow was observed. Run No. 5 had to be terminated in order to investigate the cause.

It was later found that the pressure reducer PCV-26 (after the pressure control valve PV-1) was malfunctioning. This reducer was returned to the supplier for replacement.

## 5.5 Redesign of the Hydrogen Preheater System

After receiving Contract Amendment No. 2 from the DSS, work was started immediately to design and procure the modified hydrogen preheater system. The integrated preheater system (Dwg. 100-43-SK1) consists of:

- A single-phase, phase-angle fired silicon controlled rectifier (SCR) power controller, rated 140 amps, 33.3 kVa at 240V; complete with fixed AC voltage input and variable AC power output, adjustable current limiter, current transformer and NEMA-1 type enclosure.

- A transformer rated 30 kVa, single-phase, 50Hz, primary 220V, secondary 36 volts and maximum 750 amps complete with reduced capacity taps at 26 volts, 20 volts and 15 volts.

- A 3.05 m (10 ft) long heater section made of ¼" Sch. 40 Inconel 601 pipe.

- A microprocessor-based temperature controller complete with adjustable temperature set point in the range of at least 300-1100°C, temperature indicator, gain control adjustment, high-limit alarm contact output, high-limit alarm indicator, adjustable high-limit cut-off and thermocouple burn-out upscale.

- A Cr-Al Type K thermocouple of 275-1482°C range; installed on the Inconel 601 pipe just before the reactor inlet.

- An electrical isolator installed in the hydrogen supply line, about 2.44 m (8 ft) from the transformer cable cold-end connection.

Upon completion of the installation of the integrated hydrogen preheater system towards the end of October 1983, initial testing and calibration of the system were carried out. Vendor's (SCR power controller and temperature controller) representatives were at the FHP pilot unit site on November 7 & 8 , 1983 for the final testing and calibration of the new hydrogen preheater system. Helium at about 4.58 MPa (650 psig) was used as the carrier gas.

The integrated preheater system was proven to satisfy the requirements of the Kilborn Performance Specification Nos. 2 and 3 for the preheater components, i.e. controlling the preheater gas temperature to within $\pm$ 10°C of the set temperature.

Highlights of the test results were:

- Once the SCR power controller was fired and the temperature controller set, the preheater pipe wall temperature would reach the desired temperature range in a few minutes; while at about 850°C, the preheater pipe developed a red hot colour through the length of the full-current heating section.

- The electrical current by-pass installed on the thermocouple/ reactor entrance nipple section appeared to fulfill its design purpose, i.e. lower wall temperature compared to the full-current section.

- At a low helium flow rate of 1 scfm or 28.3 $dm^3$/min., the carrier gas temperature was limited to a maximum of 450°C while the preheater pipe wall temperature was 844°C.

- At higher helium flow rates of 4-7 scfm or 113-198 $dm^3$/min., the set gas temperature >600°C was reached within seconds and maintained at $\pm$ 5°C of the setpoint.

- With the maximum power set at 50% and a helium flow rate of 7 scfm at 650 psig, the preheater unit was able to maintain the helium gas temperature at 700°C $\pm$ 5°C while the preheater pipe wall temperature was measured to be 857°C.

- An optical pyrometer was used to measure the preheater pipe wall temperature; typical temperature profile was 823°C near the cold-end terminal, 843°C in the middle length of the pipe, and 844°C near the in-line thermocouple. These temperatures were close to the pipe wall thermocouple reading of 857°C and served to confirm its reliability.

## 5.6 Run Nos. 6-11

After the new hydrogen preheater was installed, a total of six pilot plant runs, including five successful runs, were performed. The process conditions and duration of these runs are given in Section 5.2

For each of the six runs, the pilot unit was started-up, run and shut-down in accordance with the procedures given in a separate "Operating Manual for the Flash Hydropyrolysis Pilot Unit". Highlights were:

a. Hydrogen was supplied from three 41.5 MPa (6000 psig) hydrogen cylinders connected in parallel; initial and final cylinder pressure readings were taken to determine the total quantity of hydrogen consumed in the test run period.

b. Feed coal was Alberta Forestburg sub-bituminous "C" coal, predried and pulverized as described in Section 5.3.

c. Coal was fed from the coal feeder system using the 20-rpm auger motor and the auger with 6 turns per inch; before and after each test run, the weight of feed coal was measured in order to obtain the total quantity of coal fed.

d. At intervals of six minutes, the following readings were recorded on a prepared data sheet (see Table 7-12):

- time, h
- hydrogen mass flow, % scale
- reactor pressure, psig
- reactor inlet temperature, $^{o}C$
- preheater pipewall temperature, $^{o}C$
- coal feeder temperature, $^{o}C$
- reactor temperatures (zones 1,2,3,4), $^{o}C$
- reactor exit temperature, $^{o}C$
- char trap temperature, $^{o}C$

e. At intervals of fifteen minutes, effluent gas samples were taken and sent to the gas chromatograph laboratory for analysis, (See Appendix C for the analytical procedures).

f. Initial and final readings of the effluent gas flow totalizer were taken; these data plus the effluent gas analysis results were used to calculate the weight of effluent gas.

g. In the morning after each test run, the weights of char and two liquid products (from the water-cooled condenser and liquid-nitrogen chiller product traps) were measured and recorded on the data sheet.

h. The results from (a), (c), (f) and (g) were then used to carry out the material balance on a weight basis.

Tables 7-12 give the Item (a), (c), (d) and (g) data for Pilot Plant Run Nos. 6-11. The effluent gas analysis results for these runs are shown in Table 13.

The operating conditions for all of the test runs (except for Run No. 7) were very stable and stayed well within the acceptable ranges:

| | Reactor Temperature, °C | | Reactor Pressure, psig | |
|---|---|---|---|---|
| Run No. | Target | Operating Range | Target | Operating Range |
| 6 | 500 | 487 - 490 | 1,000 | 985 - 1,000 |
| 7* | 600 | 597 - 598 | 1,000 | 975 - 980 |
| 8 | 700 | 690 - 698 | 1,000 | 1,000 |
| 9 | 700 | 700 - 704 | 1,500 | 1,500 |
| 10 | 800 | 798 - 806 | 1,500 | 1,500 |
| 11 | 750 | 751 - 755 | 1,000 | 1,000 |

*Run No. 7 was an unsuccessful run; its calculated material balance was only 64%.

Table 14 summarizes the process conditions, test duration, hydrogen and coal feed rates, production rates of light oil & water, BTX, char and effluent gas, and the calculated material balance for all six pilot plant runs. The ranges of operating conditions, test duration, feed rates, production rates and material balance for the five successful runs (Nos. 6, 8, 9, 10 and 11) are:

- operating temperature: 500 to 800°C
- operating pressure: 1,000 to 1,500 psig (7.0 to 13.9 MPa)
- test run duration: 31 to 61 minutes
- hydrogen feed rate: 914.5 to 1,770 g/h
- coal feed rate: 720.4 to 965.6 g/h
- hydrogen/carbon ratio: 0.94 to 1.83
- light oil & water production rate: 87.3 to 201.3 g/h

- BTX & water production rate: 3.89 to 32.35 g/h
- char production rate: 368.0 to 818.9 g/h
- effluent gas production rate: 959.7 to 1,770 g/h
- material balance: 91.4 to 98.3%

The calculated material balances for the above-mentioned runs are well within the acceptable range, ± 15%, as specified in Section 5.1 Test Run Requirements. However, the calculated material balance for Run No. 7 was only 64%, which was unacceptable. The failure was due to a hydrogen leak from the preheater electrical isolater, which was replaced. Because of the lack of hydrogen, apparently pyrolysis had occurred. Tars were found in the char trap exit line, filters and liquid product traps.

## 5.7 Observations and Conclusions

From the results presented in Section 5.6, the following observations are made:

a. The present pilot unit proved to be a functional unit suitable for process developmental work on flash hydropyrolysis of coal.

b. The unit is capable of feeding coal up to 1 kg/h and hydrogen up to 2 kg/h. The ratio of hydrogen to coal can be varied from 1.0 to 5.0.

c. The unit can be operated up to 800$^{o}$C and 13.9 MPa (2000 psig), based on the results of the final test program and pressure testing. With further operating experience, the unit should be operable at higher temperatures and pressures, as long as they are within the maximum operating temperature and pressure criteria discussed in the separate "Operating Manual for the Flash Hydropyrolysis Pilot Unit".

d. The unit is capable of operating for at least 60 minutes at a coal feed rate of 1 kg/h and hydrogen feed rate of 2 kg/h.

FIGURE 2

MAXIMUM ALLOWABLE WORKING PRESSURE
VS TEMPERATURE, BASED ON 100 HR RUPTURE LIFE

PREHEATER
1/4" SCH. 40 INCONEL 601 PIPE

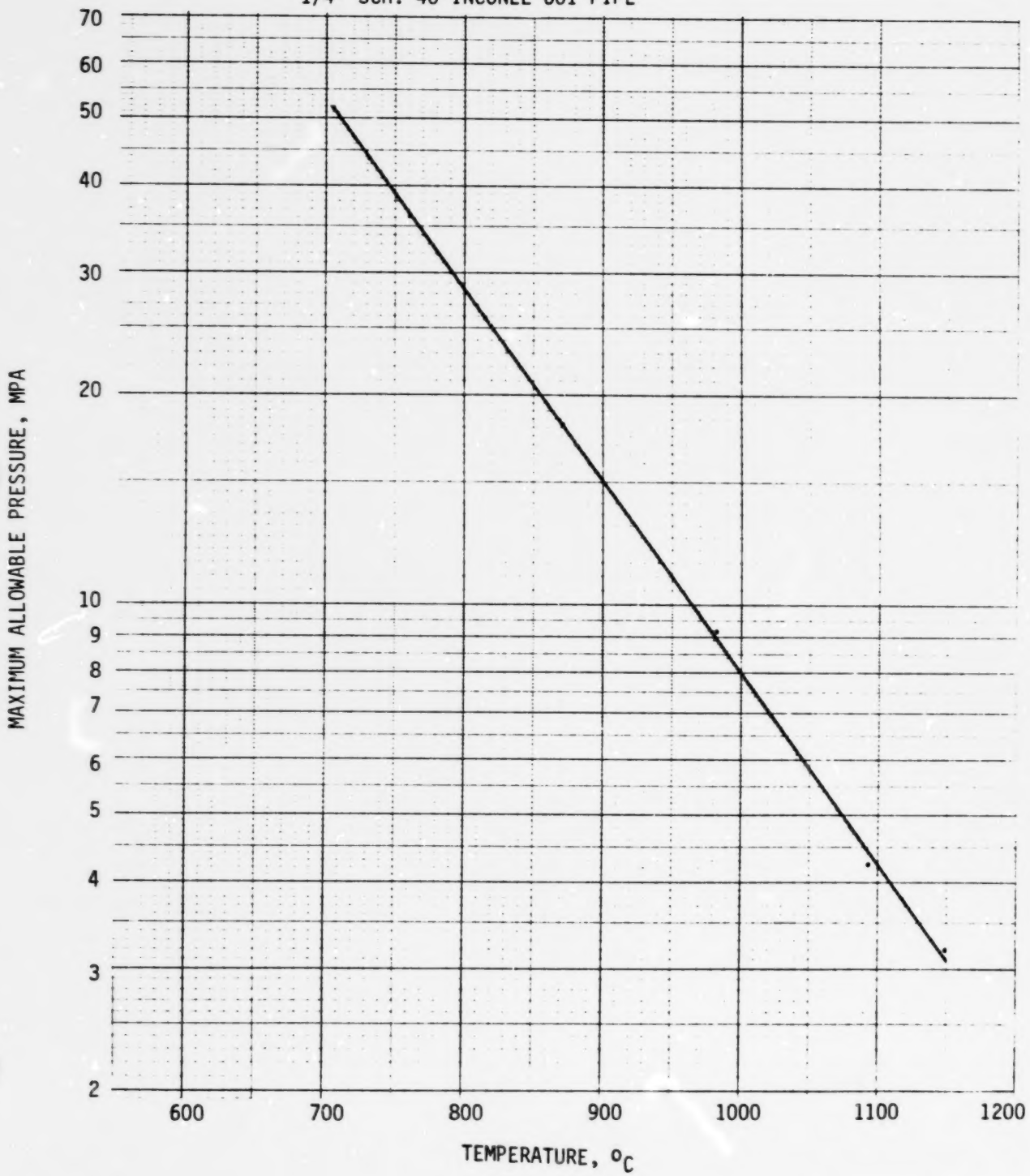
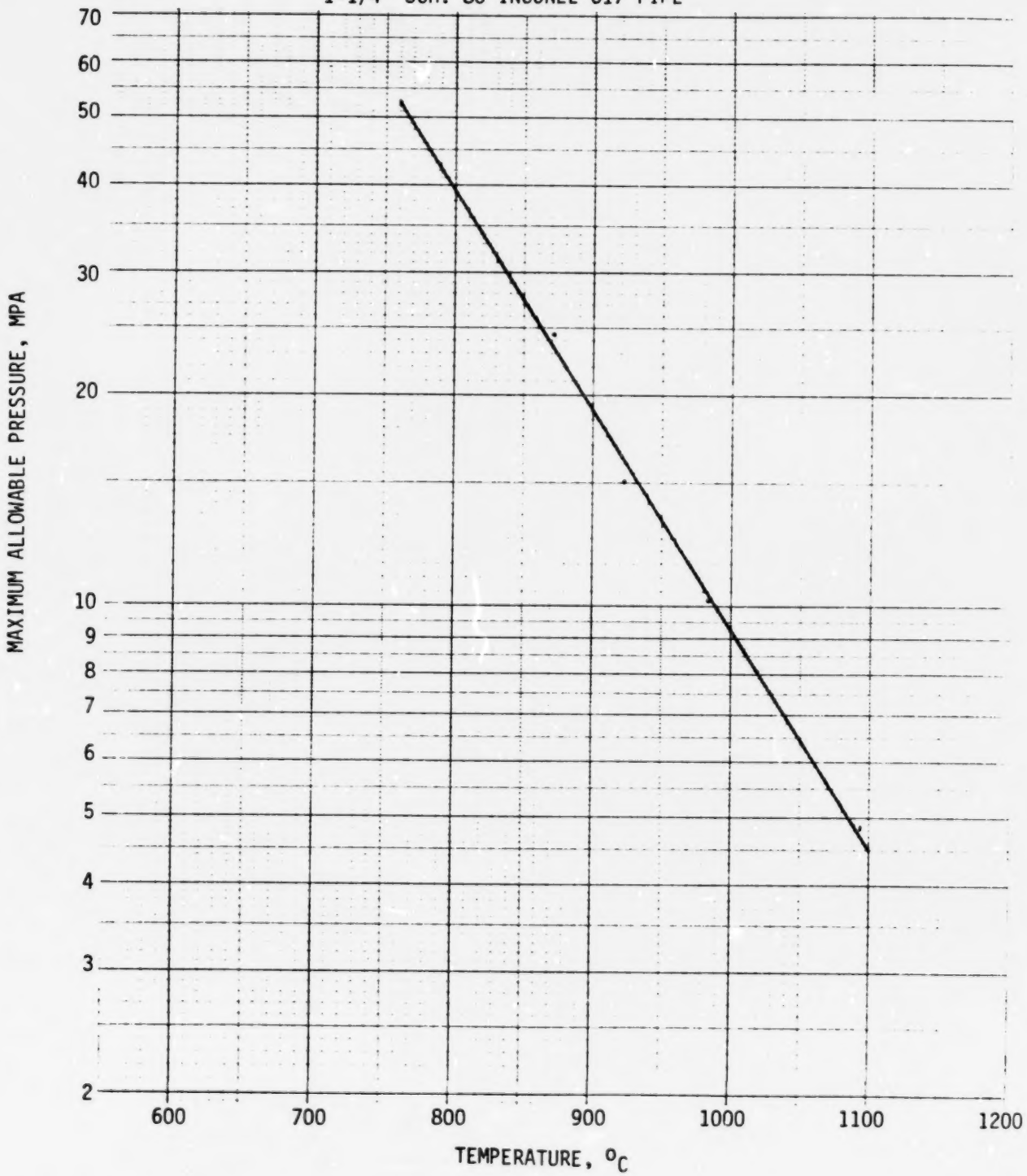


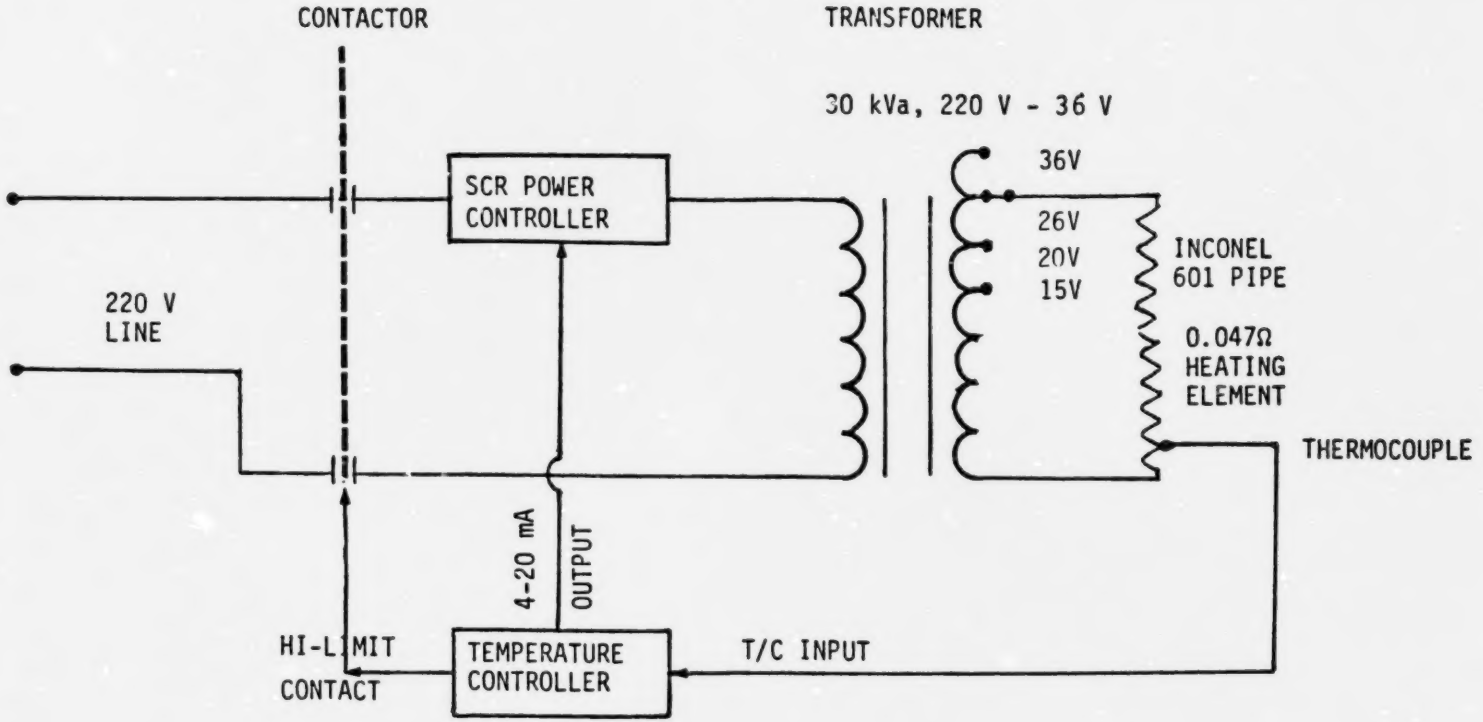
FIGURE 3

MAXIMUM ALLOWABLE WORKING PRESSURE
VS TEMPERATURE, BASED ON 100 HR RUPTURE LIFE

REACTOR

1-1/4" SCH. 80 INCONEL 617 PIPE






100-43-SK1 HYDROGEN PREHEATER SYSTEM DIAGRAM

## TABLE 6. RESULTS OF FEED COAL SAMPLE ANALYSIS

FEED COAL: Alberta Forestburg sub-bituminous "C" coal.

| A. PROXIMATE ANALYSIS | wt % |
|---|---|
| Moisture | 7.42 |
| Ash | 12.93 |
| Volatile Matter | 38.76 |
| Fixed Carbon | 40.89 |

| B. ULTIMATE ANALYSIS | wt % |
|---|---|
| C | 55.09 |
| H | 5.22 |
| N | 1.20 |
| S | 0.65 |
| Ash | 12.93 |
| Oxygen by diff. | 24.91 |

(Hydrogen and Oxygen include H and O from the sample Moisture.)

C. CALORIFIC VALUE

9,485 BTU/lb (22.06 MJ/kg)

D. HARDGROVE GRINDABILITY INDEX

36.0

E. ASH FUSION TEMPERATURE

INITIAL DEFORMATION

| | | |
|---|---|---|
| Oxidizing | 2,085°F | (1,141°C) |
| Reducing | 2,000°F | (1,093°C) |

SOFTENING TEMPERATURE

| | | |
|---|---|---|
| Oxidizing | 2,350°F | (1,288°C) |
| Reducing | 2,275°F | (1,246°C) |

HEMISPHERICAL TEMPERATURE

| | | |
|---|---|---|
| Oxidizing | 2,420°F | (1,327°C) |
| Reducing | 2,305°F | (1,263°C) |

FLUID TEMPERATURE

| | | |
|---|---|---|
| Oxidizing | 2,560°F | (1,404°C) |
| Reducing | 2,490°F | (1,366°C) |

cont'd.../2

TABLE 6 - (Cont'd.)

| F. ASH COMPOSITION | wt % |
|---|---|
| Silica | 46.98 |
| Aluminum Oxide | 20.12 |
| Iron Oxide | 4.50 |
| Calcium Oxide | 11.08 |
| Magnesium Oxide | 2.78 |
| Sodium Oxide | 4.85 |
| Potassium Oxide | 0.70 |
| Titanium Oxide | 0.86 |
| Phos. Pentoxide | 0.32 |
| Manganese Dioxide | 0.02 |
| Sulphur Dioxide | 5.72 |

| G. COAL COMPOSITION* | wt % |
|---|---|
| C | 55.09 |
| H | 4.40 |
| N | 1.20 |
| S | 0.65 |
| O | 18.31 |
| Ash | 12.93 |
| Moisture | 7.42 |

*In this composition, hydrogen and oxygen in the moisture are separated from the hydrogen and oxygen bound in the coal.

TABLE 7. FHP PILOT PLANT RUN NO. 6 RECORD



TARGET CONDITIONS: 500°C, 1000 psi

DATE: November 28, 1983.

| PARAMETER \ READING NO. | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Time | h | 12:26 | 12:35 | 12:38 | 12:48 | 15:22 | 15:28 | 15:34 | 15:39 | 15:43 | 15:47 |
| Feed Material | | He | $H_2$ | $H_2$ + coal → | | $H_2$ | $H_2$ + coal → | | | | |
| Gas Mass Flow ① [a] | % scale | 8.5 | 68.0 | 66.5 | 84.5 | 54.0 | 88.0 | 87.5 | 87.5 | 87.5 | 87.5 |
| Reactor Pressure ② | psig | 62 | 875 | 985 | 1000 | 900 | 985 | 985 | 990 | 990 | 990 |
| Reactor Inlet Temp. ③ | °C | 513 | 521 | 513 | 502 | 512 | 502 | 502 | 500 | 500 | 499 |
| Preheater Pipe Temp. | | | | | | | | | | | |
| Pipewall Pt.#1 ④ | °C | 492 | 454 | 435 | 388 | 435 | 401 | 392 | 389 | 388 | 387 |
| Pipewall Pt.#2 ⑤ | °C | 527 | 500 | 475 | 448 | 483 | 464 | 455 | 452 | 450 | 448 |
| Coal Feeder Temp. ⑥ | °C | 11 | 23 | 18 | 18 | 21 | 19 | 20 | 20 | 21 | 21 |
| Reactor Temp. | | | | | | | | | | | |
| Zone 1 ⑦ | °C | 434 | 482 | 485 | 489 | 488 | 490 | 491 | 491 | 491 | 491 |
| Zone 2 ⑧ | °C | 469 | 484 | 485 | 488 | 487 | 487 | 488 | 488 | 488 | 488 |
| Zone 3 ⑨ | °C | 480 | 490 | 490 | 492 | 491 | 491 | 491 | 491 | 491 | 491 |
| Zone 4 ⑩ | °C | 475 | 488 | 489 | 491 | 491 | 491 | 491 | 491 | 491 | 491 |
| Average | °C | 464 | 486 | 487 | 490 | 489 | 490 | 490 | 490 | 490 | 490 |
| Reactor Exit Temp. ⑪ | °C | 300 | 310 | 350 | 408 | 300 | 355 | 380 | 390 | 394 | 398 |
| Char Trap Temp. ⑫ | °C | 20 | 20 | 45 | 110 | 50 | 78 | 110 | 132 | 145 | 163 |
| Remarks | | b | | c | d,e | | f | | | g | h-l |

a. Numbers in ⬡ refer to the monitoring points shown on Dwg. 100-43-003.
b. After this reading hydrogen flow was started; helium was purged from the FHP unit.
c. Coal feeder was started at 12:37 h.
d. Hydrogen flow rate was increased at 12:45[h], from a set point of 66.5% to 84.5%
e. After a 12-min. run the FHP unit was shut down automatically, triggered by a minor hydrogen leak from the downstream connection of the effluent gas flow totalizer. Cause of the leak was corrected.

cont'd.../2

TABLE 7. FHP PILOT PLANT RUN NO. 6 RECORD



f. Coal feeder was restarted at 15:28 h. Initial hydrogen cylinder pressure was 5,300 psig.
g. Product gas samples were taken at 15:43 and 15:47 h.
h. Coal feeder was stopped at 15:50 h, after a 22-min. run in the second part of Run No. 6.
i. Reading of the effluent gas flow totalizer was 200 cu.ft. for a period of 16 minutes and 20 seconds in the second part of Run No. 6.
j. Pressure drop in the hydrogen cylinder = 5,300 - 1,150 = 4,150 psig for a period of 22 minutes in the second part of Run No. 6.
k. Total quantity of coal fed for 34 min. Run No. 6 = 547.18 g.
l. Char produced = 464.0 g, light oils & water produced = 49.46 g, BTX & water produced = 7.62 g.

TABLE 8. FHP PILOT PLANT RUN NO. 7 RECORD 

TARGET CONDITIONS: 600°C, 1000 psi DATE: December 9, 1983.

| PARAMETER \ READING NO. | | 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|---|---|
| Time | h | 12:43 | 12:53 | 13:13 | 13:27 | 13:33 | 13:40 |
| Feed Material | | He → | | $H_2$ + coal → | | | |
| Gas Mass Flow ⟨1⟩ [a] | % scale | 14-24 | 50.4 | 66.0 | 69.5 | 70.0 | 69.5 |
| Reactor Pressure ⟨2⟩ | psi | 1,020 | 715 | 975 | 980 | 980 | 980 |
| Reactor Inlet Temp. ⟨3⟩ | °C | 361 | 480 | 603 | 600 | 600 | 600 |
| Preheater Pipe Temp. | | | | | | | |
| Pipewall Pt.#1 ⟨4⟩ | °C | 462 | | 522 | 541 | 542 | 543 |
| Pipewall Pt.#2 ⟨5⟩ | °C | 498 | 652 | 574 | 585 | 587 | 588 |
| Coal Feeder Temp. ⟨6⟩ | °C | 14 | | 21 | 21 | 22 | 22 |
| Reactor Temp. | | | | | | | |
| Zone 1 ⟨7⟩ | °C | 592 | | 597 | 598 | 599 | 599 |
| Zone 2 ⟨8⟩ | °C | 591 | | 594 | 594 | 594 | 594 |
| Zone 3 ⟨9⟩ | °C | 596 | | 599 | 599 | 599 | 599 |
| Zone 4 ⟨10⟩ | °C | 596 | | 598 | 598 | 598 | 598 |
| Average | °C | 594 | | 597 | 597 | 598 | 598 |
| Reactor Exit Temp. ⟨11⟩ | °C | 260 | 380 | 445 | 465 | 465 | 465 |
| Char Trap Temp. ⟨12⟩ | °C | 16 | 22 | 50 | 78 | 86 | 86 |
| Remarks | | b | | c | d | e | f-j |

a. Numbers in ⟨⟩ refer to the monitoring points shown in on Dwg. 100-43-003.

b. Flow rate of helium was adjusted. At 12:50h the power supply to the preheater was shut off by the high-temperature cut-off being set at 600°C. This set point was raised to 650°C; the preheater was restarted immediately.

cont'd.../2

TABLE 8. FHP PILOT PLANT RUN NO. 7 RECORD



c. Hydrogen flow was started at 13:08h. Coal feeder was started at 13:12h. Initial hydrogen cylinder pressure was 4,000 psig.

d. According to the totalizer, the flow rate of the effluent gas was decreasing gradually. At 13:25h the set point of the hydrogen flow controller was increased to 69.5% from 66.0%. This resulted in activation of the hydrogen detector warning light. Product gas sample #1 was taken after the flow resetting.

e. At 13:30h product gas sample #2 was taken.

f. At 13:42h coal feeder was stopped. This completed Run No. 7 for a duration of 30 minutes and 16 seconds.

g. Total flow of the effluent gas for the period from 13:13h to 14:41h according to the totalizer, was 104 cu.ft. This included 55 cu.ft. from 13:13h to 13:25h and 49 cu.ft. from 13:26h to 13:41h.

h. Pressure drop in the hydrogen cylinder = 4,600 - 3,100 = 900 psig for a period of 30 minutes.

i. Total quantity of coal feed for 30 min. Run No. 7 = 482.82 g.

j. Char produced = 291.0 g, light oils & water produced = 32.73 g, tar produced = 20.0 g.

TABLE 9. FHP PILOT PLANT RUN NO. 8 RECORD



TARGET CONDITIONS: 700°C, 1000 psi

DATE: December 8, 1983.

| PARAMETER \ READING NO. | | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|---|
| Time | h | 10:43 | 10:53 | 11:14 | 11:30 | 11:44 |
| Feed Material | | $H_2$ → | → | $H_2$ + coal → | → | → |
| Gas Mass Flow ① [a] | % scale | 25 | 55 | 50 | 51 | 51 |
| Reactor Pressure ② | psi | 650 | 960 | 1000 | 1000 | 1000 |
| Reactor Inlet Temp. ③ | °C | 416 | 705 | 700 | 700 | 700 |
| Preheater Pipe Temp. | | | | | | |
| Pipewall Pt.#1 ④ | °C | 379 | 666 | 641 | 633 | 635 |
| Pipewall Pt.#2 ⑤ | °C | 439 | 715 | 695 | 689 | 693 |
| Coal Feeder Temp. ⑥ | °C | 17 | 22 | 23 | 26 | 27 |
| Reactor Temp. | | | | | | |
| Zone 1 ⑦ | °C | 671 | 664 | 685 | 663 | 662 |
| Zone 2 ⑧ | °C | 697 | 698 | 698 | 698 | 698 |
| Zone 3 ⑨ | °C | 703 | 704 | 704 | 704 | 697 |
| Zone 4 ⑩ | °C | 703 | 703 | 703 | 704 | 703 |
| Average | °C | 694 | 692 | 698 | 692 | 690 |
| Reactor Exit Temp. ⑪ | °C | 335 | 440 | 485 | 525 | 525 |
| Char Trap Temp. ⑫ | °C | 10 | 15 | 100 | 165 | 185 |
| Remarks | | b | c | d | e | f-i |

a. Numbers in ⬡ refer to the monitoring points shown on Dwg. 100-43-003.

b. Hydrogen flow was started at 10:43h. Initial hydrogen cylinder pressure was 5,350 psig.

c. Set point of the hydrogen flow controller was lowered to 50% at 11:02h.

d. Coal feeder was started at 11:13h. Effluent gas flow totalizer was set at zero reading at the same time.

cont'd.../2

TABLE 9. FHP PILOT PLANT RUN NO. 8 RECORD



e. Product gas sample #1 was taken at 11:23h, #2 taken at 11:33h.

f. Coal feeder was stopped at 11:44h, after a feeding period of 30 minutes and 52 seconds. Final reading of the effluent gas flow totalizer was 180 cu.ft.

g. Pressure drop in the hydrogen cylinder = 5,350 - 3,800 = 1,550 psig for a period of 60 minutes and 52 seconds.

h. Total quantity of coal fed for 30 min. Run No. 8 = 472.0 g

i. Char produced = 272.2 g, light oils & water produced = 72.1 g, BTX & water produced = 2.0 g.

TABLE 10. FHP PILOT PLANT RUN NO. 9 RECORD



TARGET CONDITIONS: 700°C, 1500 psi

DATE: December 14, 1983.

| PARAMETER \ READING NO. | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|---|
| Time | h | 13:27 | 13:35 | 13:50 | 13:59 | 14:02 | 14:10 | 14:17 | 14:24 | 14:33 |
| Feed Material | | He → | | $H_2$ | $H_2$ + coal → | | | | | |
| Gas Mass Flow ① a | % scale | 50.5 | 54.5 | 58.0 | 60.0 | 60.0 | 60.0 | 60.0 | 60.0 | 60.0 |
| Reactor Pressure ② | psi | 1190 | 1070 | 1480 | 1500 | 1500 | 1500 | 1500 | 1500 | 1500 |
| Reactor Inlet Temp. ③ | °C | 589 | 709 | 708 | 699 | 699 | 699 | 699 | 699 | 708 |
| Preheater Pipe Temp. | | | | | | | | | | |
| Pipewall Pt.#1 ④ | °C | 523 | | 632 | 625 | 619 | 613 | 611 | 607 | 620 |
| Pipewall Pt.#2 ⑤ | °C | 584 | 754 | 694 | 681 | 681 | 681 | 681 | 681 | 694 |
| Coal Feeder Temp. ⑥ | °C | 17 | 17 | | 24 | 25 | 26 | 28 | 29 | 31 |
| Reactor Temp. | | | | | | | | | | |
| Zone 1 ⑦ | °C | 673 | 681 | | 706 | 703 | 693 | 690 | 689 | 689 |
| Zone 2 ⑧ | °C | 699 | 700 | | 700 | 700 | 700 | 700 | 700 | 707 |
| Zone 3 ⑨ | °C | 704 | 705 | | 705 | 705 | 705 | 705 | 705 | 705 |
| Zone 4 ⑩ | °C | 705 | 705 | | 705 | 705 | 705 | 705 | 705 | 705 |
| Average | °C | 695 | 698 | | 704 | 703 | 701 | 700 | 700 | 702 |
| Reactor Exit Temp. ⑪ | °C | 420 | 470 | 530 | 545 | 555 | 568 | 570 | 570 | 580 |
| Char Trap Temp. ⑫ | °C | | | | | | | | | |
| Remarks | | | b | c | d | | e | | f | g |

a. Numbers in ⬡ refer to the monitoring points shown on Dwg. 100-43-003.

b. Hydrogen flow was started at 13:37h.

c. Immediately after this reading, the flow rate of hydrogen was increased from a set point of 58% to 60%. Hydrogen cylinder pressure at 13:57h was 5,150 psig.

d. Coal feeder was started at 13:58h, reading of the effluent gas totalizer was 240 cu.ft.

e. Product gas sample #1 was taken at 14:12 h.

cont'd.../2

TABLE 5. FHP PILOT PLANT RUN NO. 9 RECORD



TARGET CONDITIONS: 700°C, 1500 psi

DATE: December 14, 1983.

| PARAMETER \ READING NO. | | 10 | 11 | 12 |
|---|---|---|---|---|
| Time | h | 14:40 | 14:50 | 14:56 |
| Feed Material | | $H_2$ + coal | ——— | ——— |
| Gas Mass Flow (1) | % scale | 60.0 | 60.0 | 60.0 |
| Reactor Pressure (2) | psi | 1500 | 1500 | 1500 |
| Reactor Inlet Temp. (3) | °C | 712 | 711 | 711 |
| Preheater Pipe Temp. | | | | |
| Pipewall Pt.#1 (4) | °C | 625 | 625 | 624 |
| Pipewall Pt.#2 (5) | °C | 699 | 699 | 699 |
| Coal Feeder Temp. (6) | °C | 32 | | 34 |
| Reactor Temp. | | | | |
| Zone 1 (7) | °C | 692 | 695 | 695 |
| Zone 2 (8) | °C | 708 | 708 | 704 |
| Zone 3 (9) | °C | 705 | 705 | 705 |
| Zone 4 (10) | °C | 705 | 705 | 705 |
| Average | °C | 703 | 703 | 702 |
| Reactor Exit Temp. (11) | °C | 580 | 580 | 580 |
| Char Trap Temp. (12) | °C | | | 245 |
| Remarks | | | h | i-l |

f. Product gas sample #2 was taken at 14:28h.
g. Prior to this reading, the set points of the preheater and reactor zone #2 furnace were raised in order to offset the heat loss in reactor zone #1.
h. Product gas sample #3 was taken at 14:48h.
i. Coal feeder was stopped at 14:58h. Total feeding duration was 60 minutes and 22 seconds. Final reading of the effluent gas flow totalizer was 600 cu.ft.
j. Pressure drop in the hydrogen cylinder = 5,150 - 3,550 = 1,600 psig for a period of 59 minutes and 14 seconds.
k. Total quantity of coal fed for 60 min. Run No. 9 = 881.89 g.
l. Char produced = 447.32 g, light oils & water produced = 204.1 g, BTX & water produced = 14.51 g.

TABLE 11. FHP PILOT PLANT RUN NO. 10 RECORD



TARGET CONDITIONS: 800°C, 1500 psi

DATE: December 15, 1983.

| PARAMETER \ READING NO. | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Time | h | 15:07 | 15:18 | 15:26 | 15:37 | 15:42 | 15:53 | 15:59 | 16:05 | 16:10 | 16:16 |
| Feed Material | | He → | | $He-H_2$ | $H_2$ → | | | $H_2$ + coal → | | | |
| Gas Mass Flow (1) [a] | % scale | 55.0 | 55.0 | 61.0 | 61.0 | 61.0 | 68.5 | 68.5 | 68.2 | 68.2 | 69.0 |
| Reactor Pressure (2) | psi | 1010 | 1075 | 1300 | 1475 | 1490 | 1500 | 1500 | 1500 | 1500 | 1500 |
| Reactor Inlet Temp. (3) | °C | 664 | 774 | 815 | 824 | 844 | 833 | 807 | 814 | 828 | 840 |
| Preheater Pipe Temp. | | | | | | | | | | | |
| Pipewall Pt.#1 (4) | °C | 656 | 809 | 818 | | 843 | 816 | 774 | 787 | 811 | 833 |
| Pipewall Pt.#2 (5) | °C | 711 | 865 | 872 | 818 | 826 | 812 | 770 | 780 | 807 | 829 |
| Coal Feeder Temp. (6) | °C | 16 | 17 | 26 | 24 | 24 | 26 | 28 | 30 | 31 | 32 |
| Reactor Temp. | | | | | | | | | | | |
| Zone 1 (7) | °C | 724 | 743 | 770 | 787 | 790 | 797 | 797 | 785 | 776 | 770 |
| Zone 2 (8) | °C | 809 | 821 | 828 | 817 | 790 | 810 | 808 | 808 | 803 | 803 |
| Zone 3 (9) | °C | 810 | 810 | 810 | 810 | 810 | 810 | 810 | 810 | 810 | 810 |
| Zone 4 (10) | °C | 810 | 809 | 809 | 809 | 809 | 809 | 809 | 809 | 809 | 809 |
| Average | °C | 788 | 796 | 804 | 806 | 807 | 807 | 806 | 803 | 800 | 798 |
| Reactor Exit Temp. (11) | °C | 480 | 530 | 560 | 575 | 580 | 580 | 592 | 610 | 620 | 625 |
| Char Trap Temp. (12) | °C | 25 | 50 | 75 | 110 | 125 | 162 | 192 | 205 | 210 | 238 |
| Remarks | | b | | c | d | | e,f | g | h | | i |

a. Numbers in ⟨⟩ refer to the monitoring points shown on Dwg. 100-43-003.

b. Helium cylinder was changed at 15:10h. Before this change the temperature in reactor zone #1 was above 800°C.

c. Hydrogen flow was started at 15:25h.

d. Hydrogen flow rate was increased at 15:35h, from a set point of 61% to 62%.

e. Hydrogen flow rate was further increased at 15:48h to a set point of 68.5%.

f. Hydrogen cylinder pressure at 15:57h was 3,950 psig.

cont'd.../2

TABL ). FHP PILOT PLANT RUN NO. 10 RECORD



TARGET CONDITIONS: 800°C, 1500 psi

DATE: December 15, 1983.

| PARAMETER \ READING NO. | | 11 | 12 | 13 | 14 |
|---|---|---|---|---|---|
| Time | h | 16:20 | 16:26 | 16:33 | 16:40 |
| Feed Material | | $H_2$ + coal | ——— | ——— | ——— |
| Gas Mass Flow (1) | % scale | 68.5 | 68.5 | 68.8 | 68.5 |
| Reactor Pressure (2) | psi | 1500 | 1500 | 1500 | 1500 |
| Reactor Inlet Temp. (3) | °C | 850 | 841 | 841 | 841 |
| Preheater Pipe Temp. | | | | | |
| Pipewall Pt.#1 (4) | °C | 856 | 850 | 855 | 867 |
| Pipewall Pt.#2 (5) | °C | 854 | 849 | 855 | 867 |
| Coal Feeder Temp. (6) | °C | 34 | 36 | 36 | 37 |
| Reactor Temp. | | | | | |
| Zone 1 (7) | °C | 771 | 773 | 773 | 774 |
| Zone 2 (8) | °C | 804 | 818 | 822 | 823 |
| Zone 3 (9) | °C | 810 | 810 | 810 | 810 |
| Zone 4 (10) | °C | 809 | 809 | 809 | 809 |
| Average | °C | 799 | 803 | 804 | 804 |
| Reactor Exit Temp. (11) | °C | 625 | 625 | 625 | 625 |
| Char Trap Temp. (12) | °C | 250 | 265 | 275 | 280 |
| Remarks | | | | j | k-o |

g. Coal feeder was started at 15:58h; reading of the effluent gas flow totalizer was 1120 cu.ft.
h. Product gas sample #1 was taken at 16:02h.
i. Product gas sample #2 was taken at 16:17h.
j. Product gas sample #3 was taken at 16:32h.
k. Product gas sample #4 was taken at 16:42h.
l. Coal feeder was stopped at 16:44 h; final reading of the effluent gas flow totalizer was 1420 cu.ft. Total coal feeding duration was 45 minutes and 42 seconds.
m. Pressure drop in the hydrogen cylinder = 3,950 - 2,650 = 1,300 psig for a period of 45 minutes and 31 seconds.
n. Total quantity of coal fed for 46 min. Run No. 10 = 643.96 g.
o. Char produced = 283.8 g, light oils & water produced = 153.29 g, BTX & water produced = 18.90 g.

TABLE 12. FHP PILOT PLANT RUN NO. 11 RECORD



TARGET CONDITIONS: 750°C, 1000 psi

DATE: December 16, 1983.

| PARAMETER \ READING NO. | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|---|
| Time | h | 14:57 | 15:03 | 15:09 | 15:15 | 15:21 | 15:27 | 15:32 | 15:43 | 15:49 |
| Feed Material | | $H_2$ | ← | $H_2$ + coal | → | | | | | |
| Gas Mass Flow ① [a] | % scale | 67.5 | 67.5 | 67.4 | 67.5 | 67.5 | 67.5 | 67.8 | 68.0 | 68.3 |
| Reactor Pressure ② | psi | 975 | 980 | 1000 | 1000 | 1000 | 1000 | 1000 | 1000 | 1000 |
| Reactor Inlet Temp. ③ | °C | 750 | 752 | 750 | 748 | 748 | 748 | 748 | 752 | 752 |
| Preheater Pipe Temp. | | | | | | | | | | |
| Pipewall Pt.#1 ④ | °C | 735 | 733 | 728 | 726 | 724 | 726 | 728 | 727 | 727 |
| Pipewall Pt.#2 ⑤ | °C | 779 | 778 | 776 | 773 | 768 | 768 | 771 | 768 | 767 |
| Coal Feeder Temp. ⑥ | °C | 20 | 20 | 21 | 23 | 24 | 25 | 26 | 28 | 30 |
| Reactor Temp. | | | | | | | | | | |
| Zone 1 ⑦ | °C | 734 | 748 | 757 | 757 | 757 | 757 | 753 | 746 | 743 |
| Zone 2 ⑧ | °C | 750 | 750 | 750 | 750 | 750 | 750 | 750 | 750 | 750 |
| Zone 3 ⑨ | °C | 756 | 756 | 756 | 756 | 756 | 756 | 756 | 756 | 756 |
| Zone 4 ⑩ | °C | 756 | 756 | 756 | 756 | 756 | 756 | 756 | 756 | 756 |
| Average | °C | 749 | 752 | 755 | 755 | 755 | 754 | 753 | 752 | 751 |
| Reactor Exit Temp. ⑪ | °C | 487 | 492 | 507 | 518 | 524 | 530 | 535 | 540 | 540 |
| Char Trap Temp. ⑫ | °C | 85 | 110 | 140 | 155 | 172 | 192 | 205 | 234 | 245 |
| Remarks | | b | | c | | | d | e | | |

a. Numbers in ◯ refer to the monitoring points shown on Dwg. 100-43-003.

b. Hydrogen flow was started at 14:55h to replace helium. Preheater and four reactor furnaces were all set at 750°C. Hydrogen cylinder pressure at 14:58h was 5,600 psig.

c. Coal feeder was started at 15:09h; reading of the effluent gas flow totalizer was 300 cu.ft.

d. Product gas sample #1 was taken at 15:23h.

cont'd.../2

TABLE 12. FHP PILOT PLANT RUN NO. 11 RECORD



TARGET CONDITIONS: 750°C, 1000 psi

Date: December 16, 1983.

| PARAMETER \ READING NO. | | 10 | 11 | 12 |
|---|---|---|---|---|
| Time | h | 15:55 | 16:01 | 16:07 |
| Feed Material | | $H_2$ + coal ——→ | | |
| Gas Mass Flow (1) | % scale | 68.5 | 68.3 | 68.5 |
| Reactor Pressure (2) | psi | 1000 | 1000 | 1000 |
| Reactor Inlet Temp. (3) | °C | 752 | 752 | 752 |
| Preheater Pipe Temp. | | | | |
| Pipewall Pt.#1 (4) | °C | 726 | 725 | 726 |
| Pipewall Pt.#2 (5) | °C | 766 | 765 | 766 |
| Coal Feeder Temp. (6) | °C | 30 | 31 | 32 |
| Reactor Temp. | | | | |
| Zone 1 (7) | °C | 742 | 740 | 740 |
| Zone 2 (8) | °C | 750 | 750 | 750 |
| Zone 3 (9) | °C | 756 | 756 | 756 |
| Zone 4 (10) | °C | 756 | 756 | 756 |
| Average | °C | 751 | 751 | 751 |
| Reactor Exit Temp. (11) | °C | 540 | 540 | 540 |
| Char Trap Temp. (12) | °C | 255 | 265 | 272 |
| Remarks | | f | | g-j |

e. Product gas sample #2 was taken at 15:33h.
f. Product gas sample #3 was taken at 15:52h.
g. Coal feeder was stopped after a feeding duration of 61 minutes and 22 seconds. Final reading of the effluent gas flow totalizer was 700 cu.ft.
h. Pressure drop in the hydrogen cylinder = 5,600 - 3,570 = 2,030 psig for a period of 71 minutes.
i. Total quantity of coal fed = 736.8 g for 61 min. Run No. 11.
j. Char produced = 376.38 g, light oils & water = 153.59 g, BTX & water = 33.09 g.

TABLE 13. PRODUCT GAS ANALYSIS OF PILOT PLANT RUNS

| TEST NO. | #7 | #7 | #7 | #8 | #8 | #8 |
|---|---|---|---|---|---|---|
| SAMPLE NO. | #1 | #2 | Average | #1 | #2 | Average |
| GAS COMPOSITION, mol % | | | | | | |
| CO | 0.21 | 0.26 | 0.235 | 0.30 | 0.30 | 0.30 |
| $CO_2$ | | 0.02 | 0.01 | 0.24 | 0.03 | 0.135 |
| $CH_4$ | 0.17 | 0.75 | 0.46 | 0.63 | 0.71 | 0.67 |
| $C_2H_4$ | | 0.01 | 0.005 | 0.03 | | 0.015 |
| $C_2H_6$ | | 0.08 | 0.04 | 0.30 | 0.13 | 0.215 |
| $H_2$ | 99.62 | 98.88 | 99.25 | 98.50 | 98.83 | 98.665 |
| Total | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| WEIGHT, g/100 g-moles | | | | | | |
| C | 4.56 | 14.52 | 9.54 | 21.96 | 15.60 | 18.78 |
| H | 199.92 | 201.28 | 200.60 | 201.44 | 201.28 | 201.36 |
| O | 3.36 | 4.80 | 4.08 | 12.48 | 5.76 | 9.12 |
| Total | 207.84 | 220.60 | 214.22 | 235.88 | 222.64 | 229.26 |
| AVERAGE M.W. | 2.078 | 2.206 | 2.142 | 2.359 | 2.226 | 2.293 |

NOTE: For Test No. 6, product gas analysis indicated all components except $H_2$ to be below detectable limits (see Appendix C).

TABLE 13 - (Cont'd.)

| TEST NO. | #9 | #9 | #9 | #9 |
|---|---|---|---|---|
| SAMPLE NO. | #1 | #2 | #3 | Average |
| GAS COMPOSITION, mol % | | | | |
| CO | 0.35 | 0.33 | 0.32 | 0.333 |
| $CO_2$ | 0.016 | 0.084 | 0.175 | 0.092 |
| $CH_4$ | 0.37 | 0.8 | 0.76 | 0.643 |
| $C_2H_4$ | | | | |
| $C_2H_6$ | 0.15 | 0.42 | 0.36 | 0.310 |
| $H_2$ | 98.514 | 98.366 | 98.385 | 98.422 |
| Total | 100.00 | 100.00 | 100.00 | 100.00 |
| WEIGHT, g/100 g-moles | | | | |
| C | 12.432 | 24.648 | 23.700 | 20.256 |
| H | 199.408 | 202.452 | 201.970 | 201.276 |
| O | 6.112 | 7.968 | 10.720 | 8.272 |
| Total | 217.952 | 235.068 | 236.390 | 229.804 |
| AVERAGE M.W. | 2.180 | 2.351 | 2.364 | 2.298 |

TABLE 13 - (Cont'd.)

| TEST NO. | #10 | #10 | #10 | #10 | #10 |
|---|---|---|---|---|---|
| SAMPLE NO. | # 1 | # 2 | # 3 | # 4 | Average |
| GAS COMPOSITION, mol % | | | | | |
| CO | 0.333 | 0.35 | 0.403 | 0.399 | 0.371 |
| $CO_2$ | | | | | |
| $CH_4$ | 1.07 | 1.29 | 1.53 | 1.68 | 1.393 |
| $C_2H_4$ | | | | | |
| $C_2H_6$ | 0.12 | 0.218 | 0.24 | 0.257 | 0.209 |
| $H_2$ | 98.477 | 98.142 | 97.827 | 97.664 | 98.028 |
| Total | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| WEIGHT, g/100 g-moles | | | | | |
| C | 19.716 | 24.912 | 28.956 | 31.116 | 26.184 |
| H | 201.954 | 202.752 | 203.214 | 203.590 | 202.882 |
| O | 5.328 | 5.600 | 6.448 | 6.384 | 5.936 |
| Total | 226.998 | 233.264 | 238.618 | 241.090 | 235.002 |
| AVERAGE M.W. | 2.270 | 2.333 | 2.386 | 2.411 | 2.350 |

TABLE 13 - (Cont'd.)

| TEST NO. | #11 | #11 | #11 | #11 |
|---|---|---|---|---|
| SAMPLE NO. | # 1 | # 2 | # 3 | Average |
| GAS COMPOSITION, mol % | | | | |
| CO | 0.162 | 0.249 | 0.365 | 0.259 |
| $CO_2$ | | 0.034 | 0.031 | 0.022 |
| $CH_4$ | 0.34 | 0.555 | 0.711 | 0.535 |
| $C_2H_4$ | | | | |
| $C_2H_6$ | 0.033 | 0.156 | 0.186 | 0.125 |
| $H_2$ | 99.465 | 99.006 | 98.707 | 99.059 |
| Total | 100.00 | 100.00 | 100.00 | 100.00 |
| WEIGHT, g/100 g-moles | | | | |
| C | 6.816 | 13.800 | 17.748 | 12.792 |
| H | 200.488 | 201.168 | 201.374 | 201.008 |
| O | 2.592 | 5.072 | 6.832 | 4.848 |
| Total | 209.896 | 220.040 | 225.954 | 218.648 |
| AVERAGE M.W. | 2.099 | 2.200 | 2.260 | 2.186 |

TABLE 14. RESULTS OF FIVE SUCCESSFUL PILOT PLANT RUNS

| TEST NO.[a] | #6 | #7[b] | #8 | #9 | #10 | #11 |
|---|---|---|---|---|---|---|
| TEMPERATURE, °C | 500 | 600 | 700 | 700 | 800 | 750 |
| PRESSURE, psi | 1,000 | 1,000 | 1,000 | 1,500 | 1,500 | 1,000 |
| DURATION, min | 34 | 30 | 31 | 60 | 46 | 61 |
| REACTANTS | | | | | | |
| Hydrogen, g/h | 1,770.00 | 1,015.68 | 861.69 | 914.50 | 967.30 | 967.30 |
| Coal, g/h | 965.61 | 965.61 | 917.49 | 876.53 | 845.46 | 720.39 |
| Total, g/h | 2,735.61 | 1,981.29 | 1,779.18 | 1,791.03 | 1,812.76 | 1,687.69 |
| Hydrogen/carbon | 1.83 | 1.05 | 0.94 | 1.04 | 1.14 | 1.34 |
| PRODUCTS | | | | | | |
| Light oil & water, g/h | 87.28 | 65.46 | 133.96 | 202.86 | 201.26 | 150.17 |
| BTX, g/h | 13.45 | 40.00[c] | 3.89 | 14.42 | 24.81 | 32.35 |
| Char, g/h | 818.82 | 582.00 | 528.73 | 444.60 | 372.60 | 368.00 |
| Gas, g/h | 1,770.00 | 587.63 | 959.74 | 983.68 | 1,106.10 | 1,027.25 |
| Total, g/h | 2,689.55 | 1,275.09 | 1,626.32 | 1,645.56 | 1,704.77 | 1,577.77 |
| MATERIAL BALANCE | | | | | | |
| Σ PProd./Σ React., % | 98.3 | 64.4 | 91.4 | 91.9 | 94.0 | 93.5 |

a. Pilot plant runs, including successful and unsuccessful runs are numbered sequentially.

b. Run #7 was an unsuccessful run in terms of material balance.

c. No BTX was obtained. 40.00 g/h indicates the amount of tars collected from the lines and filters.

# APPENDIX A

## REQUIREMENTS FOR PILOT PLANT RUNS

## AS SPECIFIED BY DSS CONTRACT AMENDMENT NO. 2

DSS File No. 18SQ.23440-9-9151

Contract Serial No.: OSQ80-00019

Amendment No. 2

TITLE: DEVELOPMENT OF EQUIPMENT AND PROCESS INFORMATION RELATED TO THE FLASH HYDROPYROLYSIS OF COAL (no change)

In Appendix "A", Statement of Work:

Task 2 includes commissioning and shakedown of the bench scale flash hydropyrolysis system and a demonstration that the system is capable of operating in accordance with the specifications and requirements of the contract. Included in the shakedown operation will be at least five (5) pilot plant runs feeding a representative Canadian coal, specified by CANMET.

Each run shall commence with coal being fed into the preheated hydrogen stream. Each run shall be targetted to last one (1) hour with two (2) runs, each being a minimum of thirty (30) minutes in duration and three (3) runs each being a minimum of twenty (20) minutes in duration. After each run, the reactor and product collection traps must be depressurized and product samples collected.

The bench scale hydropyrolysis system must meet the following requirements for each pilot plant run:

(a) The coal feeder must function in accordance with Section 2 entitled "Function of the Coal Feeder".

(b) Product samples shall be collected for all output streams for each run.

(c) Reactor and hydrogen preheater temperatures shall be maintained within plus or minus 15 deg. C of setpoint.

(d) Reactor pressure control shall be maintained within plus or minus 15 psi (.103 MPa) of setpoint.

(e) Flow rate control shall be plus or minus five (5%) of minimal value.

(f) Two (2) of the runs shall be at 700 deg. C and 800 deg. C respectively at pressure of 1500 psi (10.3 MPa). The remaining three (3) runs shall be at temperatures and pressures of not less than 500 deg. C and 1000 psi (6.9 MPa). One run shall demonstrate a nominal hydrogen flow rate of 2 (two) kg per hour.

(g) The overall material balance on the system shall be demonstrated in a minimum of 5 (five) successful runs with various durations. The sum of the product weights, namely gases, entrapped liquids and chars shall be within (plus or minus) 15% of the weight of reactants (coal and hydrogen). This will require product gas analysis. Runs that fail to demonstrate the mentioned limits for material balance will not be considered successful runs.

In addition, it shall be demonstrated that the hydropyrolysis system will meet the following requirements:

1. Pressure Testing of the Unit

This involves demonstrating that the unit can hold pressure for a five hour period while the vessels (reactor and preheater) are at elevated temperature. Thus with the reactor maintained at 800°C at a pressure of 2000 psi (13.8 MPa) and the pressure shall not drop by more than 100 psi (0.69 MPa) in a five (5) hour period. Should the pressure drop by more than 100 psi within the specified period of time, the leaks shall be detected and eliminated.

2. Function of the Coal Feeder

It shall be demonstrated that the coal feeder is capable of feeding controllable amounts of coal without blockage problems. The range of coal feed rate shall be between 100 and 1000 gm/hr. Calibration between the speed of the auger motor and coal feed rate shall be established for 100 mesh particle size, Alberta Forestburg sub-bituminous coal. Should the feeder show blockage, modifications will be made to ensure proper functioning (e.g. back stream of hydrogen with positive pressure on vibrating shaft in the coal bed).

# APPENDIX B

## DATA SHEETS OF
## PILOT PLANT RUN NOS. 1-5

(January 28 - February 25, 1983)

Test #1 January , 1983

Table 1

| 1 Time hr/min | 2 Flow Tot $ft^3$ | 3 Flowrate scfm | 4 Reactor inlet °C | 5 Zone #4 °C | 6 Reactor Exit, °C | 7 Reactor psi | 8 Pre-heat Furnace °C | 9 #1 Furnace °C | 10 #2 Furnace °C | 11 #3 Furnace °C | 12 #4 Furnace °C | 13 Coal Feed °C |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 10:15 | System is filled with helium. Heaters are turned on. | | | | | | | | | | | |
| 11:30 | Pressure exceeds set value of 260 psi. Automatic safety system vents gas. | | | | | | | | | | | |
| 11:45 | Restart. Various flowrates between 1.8 and 8.4 scfm are maintained to obtain experience with controls. | | | | | | | | | | | |
| 2:52 | 1640 | 1.83 | 583 | 470 | 164 | 325 | 973 | 881 | 952 | 986 | 954 | 31 |
| 3:06 | | | 657 | 542 | 160 | 325 | 970 | 886 | 960 | 982 | 960 | |
| 3:07 | Flow of helium is shut off. | | | | | | | | | | | |
| 3:38 | 1703 | zero | 360 | 305 | 134 | 470 | 1090 | 970 | 1000 | 1000 | 979 | 33 |
| 4:00 | 1703 | zero | 330 | 273 | 125 | 450 | 1090 | 1000 | 1000 | 1000 | 980 | 33 |
| 4:30 | 1703 | zero | 328 | 262 | 112 | 448 | 1112 | 1000 | 1030 | 1033 | 981 | 34 |
| 4:50 | 1703 | zero | 338 | 255 | 104 | 448 | 1100 | 1021 | 1050 | 1060 | 1000 | 34 |
| 4:56 | 1717 | 3.80 | Flow of helium is set. | | | | | | | | | |
| | 1750 | 7.14 | | | | | | | | | | |
| 5:06 | | 7.14 | 700 | 527 | 117 | 350 | 1050 | 960 | 1012 | 1040 | 1000 | 34 |
| 5:22 | Flow of hydrogen starts. Coal feeder temperature rises to 48/49 °C, steadies at 47°C. (2,000) | | | | | | | | | | | 47 |
| 5:34 | Pressure in hydrogen bottle has dropped from 6000 to 3000 psi. Line pressure is reduced from 2000 to 1500 psi. | | | | | | | | | | | |
| 5:36 | Coal feeder and liquid nitrogen cooler are started up. (1,500) | | | | | | | | | | | |
| 5:37 | | | 632 | 680 | 192 | 1427 | 925* | 790* | 900* | 960 | 960 | 40 |
| 5:42 | | | 570 | 730 | 220 | 1455 | 900* | 760* | 870* | 940* | 950* | 38 |
| 5:47 | | | 570 | 710 | 220 | 1175 | 928 | 770 | 900 | 942 | 941 | 34 |
| 5:48 | After 12.27 min of feeding the pressure of hydrogen bottle drops to 1500 psi, hydrogen flow and coal feeder are shut off. Hydrogen is vented, system is filled with helium. | | | | | | | | | | | |
| 6:02 | 2257 | | | | | | | | | | | |

Test #2 February 3, 1983 Table 2

| 1 Time hr/min | 2 Flow Tot $ft^3$ | 3 Flowrate scfm | 4 Reactor inlet °C | 5 Zone #4 °C | 6 Reactor Exit °C | 7 Reactor psi | 8 Pre-heat Furnace °C | 9 #1 Furnace °C | 10 #2 Furnace °C | 11 #3 Furnace °C | 12 #4 Furnace °C | 13 Coal Feed °C |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 9:00 | System is filled with helium. Heaters are turned on. | | | | | | | | | | | |
| 9:10 | 2382.5 | 0 | 25 | 5 | 18 | 700 | 391 | 229 | 247 | 249 | 239 | 22 |
| 9:40 | 2382.5 | 0 | 30 | 80 | 18 | 700 | 734 | 599 | 633 | 637 | 585 | 22 |
| 10:03 | 2399 | 0 | 40 | 230 | 18 | 710 | 875 | 767 | 814 | 821 | 757 | 22 |
| 10:25 | 2399 | 0 | 40 | 212 | 19 | 710 | 960 | 858 | 911 | 924 | 853 | 23 |
| | The insulation on the pre-heater is re-adjusted to compensate for the heat expansion. | | | | | | | | | | | |
| 10:35 | 2399 | 0 | 40 | 210 | 18 | 710 | 998 | 894 | 943 | 954 | 885 | 23 |
| 11:05 | 2399 | 0 | 40 | 232 | 18 | 710 | 1116 | 978 | 1005 | 1014 | 968 | 24 |
| | All four reactor heaters reach set temperature of 1000°C at 11:28. Setting of pre-heater is advanced from 1200 to 1275 with the intention to accelerate heating. | | | | | | | | | | | |
| 11:28 | | 0 | | 247 | 18 | 710 | 1164 | 1016 | 1005 | 1014 | 1001 | 25 |
| 11:48 | Pre-heater temperature reaches 1200 °C. The controls are set to 1200 °C, the heater cuts out, the temperature drops almost instantaneously to 1177 °C. Preparations for the conversion to "flow" condition are in progress. | | | | | | | | | | | |
| 12:35 | Pressure is dropping at a fast rate - leak. Many warning lights, continuous cut in/cut out noises from control panel. The system is shut off. "Gas Line Pressure" light only is lit. Coal feeder temperature drops from 28 to 15 °C. Test is terminated. | | | | | | | | | | | |
| 1:05 | 2399 | 0 | | 160 | 18 | 0 | 733 | 698 | 738 | 744 | 676 | 26 |

| 1 Time hr/min | 2 Flow Tot $ft^3$ | 3 Flowrate scfm | 4 Reactor inlet °C | 5 Zone #4 °C | 6 Reactor Exit °C | 7 Reactor psi | 8 Pre-heat Furnace °C | 9 #1 Furnace °C | 10 #2 Furnace °C | 11 #3 Furnace °C | 12 #4 Furnace °C | 13 Coal Feed °C |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 8:30 | System is put on. Pressure reducer upstream totalizer has been removed, replacement regulator has been sent for. | | | | | | | | | | | |
| 8:45 | 2432.5 | 0 | U/S | | | | 672 | 438 | 489 | 476 | 477 | |
| 9:02 | 2432.5 | 0 | U/S | 100 | 15 | 312 | 826 | 646 | 676 | 678 | 625 | 25 |
| | A low flowrate is set to check Reactor Inlet Temp. thermocouple. Flow is shut off. | | | | | | | | | | | |
| 9:15 | | 0 | | | | 300 | 917 | 757 | 791 | 893 | 734 | |
| 9:30 | | 0 | U/S | 180 | | 300 | 987 | 837 | 870 | 873 | 809 | 25 |
| 10:00 | | 0 | 200 | 275 | | 325 | 991 | 919 | 947 | 947 | 891 | 25 |
| 10:30 | | 0 | 140 | 250 | 15 | 325 | 999 | 950 | 950 | 949 | 951 | 26 |
| | All furnaces have reached the set temperatures in 2 hours. | | | | | | | | | | | |
| 11:00 | | | 140 | 250 | 20 | 300 | 999 | 950 | 950 | 950 | 950 | 26 |
| 11:44 | 2435 | 0 | 100 | 260 | 15 | 280 | 1000 | 950 | 950 | 950 | 950 | 28 |
| 11:50 | | 0 | 275 | 350 | | | 997 | 941 | 949 | 950 | 949 | |
| | Experiments to set 4.9 scfm He start - 3.2 scfm is the uniform rate from 11:55 on. Flowtest 3/1. | | | | | | | | | | | |
| 12:00 | 2465 | 3.2 | 430 | 450 | | 550 | 997 | 924 | | | | |
| 12:02 | | 3.2 | 550 | 520 | | | 999 | 922 | 950 | 950 | 950 | |
| 12:13 | 2495 | 3.2 | 552 | 552 | 20 | 552 | 1000 | 920 | 950 | 950 | 950 | 26 |
| | Flow increases to 9.1 scfm. | | | | | | | | | | | |
| 12:16 | 2527 | 9.1 | 650 | 575 | 40 | | 998 | 917 | 951 | 950 | 951 | 28 |
| 12:19 | 2551 | Flow is shut-off, bottle exhausted. New bottle installed. | | | | | | | | | | |
| 12:35 | | 0 | 425 | 560 | | | 1000 | 950 | 950 | 950 | 950 | 29 |
| 12:50 | A o $ft^3$ "spill" into the reactor as new bottle is connected. Normal flow starts at 12:55. Flowtest 3/2 | | | | | | | | | | | |
| 12:53 | | | 520 | 480 | | 1,000 | 952 | 931 | 948 | 949 | 950 | 42 |
| 12:58 | 2588 | 8.2 | 630 | 530 | | | 968 | 914 | 949 | 949 | 950 | 31 |
| 1:00 | 2608 | 8.2 | 665 | 575 | | | 965 | 902 | 949 | 949 | 950 | 31 |
| 1:05 | | 8.2 | 685 | 610 | 58 | 1005 | 967 | 880 | 949 | 949 | 950 | 31 |

| 1 Time hr/min | 2 Flow Tot $ft^3$ | 3 Flowrate scfm | 4 Reactor inlet °C | 5 Zone #4 °C | 6 Reactor Exit °C | 7 Reactor psi | 8 Pre-heat Furnace °C | 9 #1 Furnace °C | 10 #2 Furnace °C | 11 #3 Furnace °C | 12 #4 Furnace °C | 13 Coal Feed °C |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1:09 | 2699 | 8.1 | 690 | 630 | | | 963 | 872 | 949 | 950 | 950 | 29 |
| | Bottle is exhausted; flow stops. Sample lines purged, He is exhausted from reactor. | | | | | | | | | | | |
| 1:14 | | 0 | 540 | 560 | | | 986 | 907 | 952 | 951 | 951 | 31 |
| 1:23 | | 0 | 450 | 510 | | | 996 | 944 | 949 | 950 | 951 | 31 |
| 1:40 | Line from pre-heater to reactor is covered by insulating blanket. | | | | | | | 1:45 Pre-heater set 1000 →1050 | | | | |
| 1:45 | | 0 | 270 | 470 | | | 998 | 950 | 949 | 950 | 951 | 30 |
| 1:50 | | 0 | 250 | 410 | | | 1035 | 950 | 950 | 950 | 950 | 31 |
| 2:00 | 2723 | 0 | 215 | 380 | 62 | 360 | 1046 | 950 | 950 | 950 | 950 | 31 |
| 2:10 | 2723 | 0 | 190 | 360 | | | 1049 | 950 | 950 | 950 | 950 | |
| 2:11 | Helium flow is on. Coal feeder temperature shuts up to 61 °C. | | | | | | | | Flowtest 3/3 | | | 61 |
| 2:15 | 2732 | 5.4 | 490 | 520 | | /500 | 954 | 924 | 949 | 950 | 950 | 41 |
| 2:16 | Bottle is exhausted, flow stops. | | | | | | | | | | | |
| 2:23 | 2759 | 0 | 500 | 580 | | | 1040 | 932 | 951 | 951 | 950 | 36 |
| 2:26 | With helium shut-off, hydrogen about to be connected, pressure is lost. Test is terminated. | | | | | | | | | | | |

TEST # 4 FEBRUARY 18, 1983 TABLE 4 1 of 2

| 1 Time | 2 Totaliz. | 3 Flow-rate | 4 Reactor inlet | 5 Zone #4 | 6 Reac exit | 7 for press. | 8 Pre-heat F#1 | 9 Coal F. C#1 | 10 #1 Furn C#2 | 11 #2 Furn C#3 | 12 #3 Furn. C#4 | 13 #4 Furn. C#5 | 14 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| hr:min | ft3 | | °C | °C | °C | psi | °C | °C | °C | °C | °C | °C | |
| 7:00 | Check: Some of the electrical connections to the glowbars in the pre-heater are burned out. | | | | | | | | | | | | |
| | | Some leakage in the system. | | | | | | | | | | | |
| | There | are corrected. | System | is tested with $N_2$ | | | | to save on cost of He | | | | | |
| 10:15 | Change to | He flow. | Adjusting rates: | | | | | | | | | | |
| 10:23 | 2912 | 4.68 | 370 | 320 | 25 | 285 | 680 | 23 | 538 | 602 | 618 | 612 | |
| 10:36 | 2980 | 1.73 | | | | | | | | | | | |
| 10:40 | 2986 | .39 | 370 | 405 | 37 | 263 | 830 | 23 | 691 | 736 | 756 | 729 | |
| 10:50 | 2989 | 1.03 | This | rate is | accepted | for | the heating up | process | | | | | |
| 10:54 | 2993 | -11- | 280 | 400 | 37 | 285 | 845 | 23 | 770 | 817 | 835 | 799 | |
| 10:58 | The gas line | directly | into the | coal feeder (by-passing pre-heater) | | | | is | shut | off. | | | |
| 11:01 | 3000 | 1.08 | 270 | 420 | 38 | 290 | 848 | 23 | 795 | 850 | 850 | 827 | |
| 11:26 | All heaters are at the set 850°C temperature. The pre-heater maintains it with modulation, | | | | | | | | | | | | |
| | the reactor heaters with on/off. Pre-heater is set to 860 °C at 11:38 to check power. | | | | | | | | | | | | |
| | 3026 | 1.05 | 250 | 457 | 37 | 295 | 848 | 24 | 850 | 850 | 850 | 850 | |
| 11:43 | 3043 | 1.03 | 245 | 460 | 37 | 300 | 856 | 24 | 850 | 850 | 850 | 850 | |
| 11:53 | Checking/preparation for $H_2$ start. | | | | | Two $H_2$ bottles in parallel at 5100 psi, ± 5°C | | | | | | | |
| 12:03 | 3066 | 1.02 | 245 | 460 | 37 | 290 | 860 | 24 | 850 | 850 | 850 | 850 | |
| 12:07 | 3070 | 1.02 | Reactor | room | door | is | closed. | | | | | | |
| 12:15 | Liquid $N_2$ is | set | initially | to | 0°F. | $H_2$ is | on. | | | | | | |
| 12:16 | System is | laboriously | pressurized. | | | | | 30 | | | | | |
| 12:22 | 3091 | 9.26 | | | | | | | | | | | |
| 12:24 | | | 560 | 600 | 88 | 750 | 833 | 30 | 800 | 850 | 850 | 850 | |
| 12:28 | 3167 | 4.95 | Bottles | 4250/1000 psi. | Reducing | flowrate | laboriously. | | | | | | |
| 12:34 | Reducing flowrate with | controller, | increasing | line | pressure | in | compressor | heard. | | | | | |
| 12:35 | | | 555 | 660 | 145 | 1500 | 808 | 40 | 791 | 850 | 850 | 850 | |

B-5

TEST #4 FEBRUARY 18, 1983 TABLE 4

| 1 Time | 2 Totaliz. | 3 Flow-rate | 4 Reactor inlet | 5 Zone #4 | 6 Reactor exit | 7 press. | 8 Pre-heat F#1 | 9 Coal F. C#1 | 10 #1 Furn C#2 | 11 #2 Furn C#3 | 12 #3 Furn C#4 | 13 #4 Furn. C#5 | 14 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| hr:min | ft³ | | °C | °C | °C | psi | °C | °C | °C | °C | °C | °C | |
| 12:37 | Re-set | pre-heater to | | 900°C | in order to | | maximize heating power | | | | | | |
| 12:40 | 3246 | 6.47 | | | | | | | | | | | |
| 12:45 | | | 580 | 750 | 170 | 1925 | 811 | 34 | 753 | 850 | 850 | 850 | |
| 12:48 | Re-set | zones | #2, 3, 4 | to | 800°C | to prevent | overheating | | | | | | |
| 12:50 | 3313 | 7.60 | Bottles | 3000/2100 | | This | is the | minimum | flowrate | possible | with | controller. | |
| 12:54 | 3344 | 7.50 | " | 2800/2100 | psi | | | | | | | | |
| 12:57 | Coal | feeder | on | | | | | | | | | | |
| 12:58 | | | 598 | 750 | 190 | 1940 | 855 | 33 | 754 | 800 | 800 | 800 | |
| 1:01 | 3377 → 3381 | | Bottles | 2550/2100 | psi | seconds | before | the | explosion. | | | | |
| | | ← stopped at | this | value | after | the | explosion. | | | | | | |
| | Sound and flash of | | a minor | explosion | in | heard. | Leakage | noted, | shut | down. | The | gasket | |
| | on the | bottom | end of | the | reactor | cooler | was | blown. | Temp. | of | liquid | $N_2$ | reached - 50°F = -65°C. |

B-6

TEST #5 FEBRUARY 25, 1983 TABLE 5



| 1 Time | 2 Totaliz. | 3 Flow-rate | 4 Reactor inlet | 5 Zone #4 | 6 Reactor exit | 7 press. | 8 Pre-heat F#1 | 9 Coal F. C#1 | 10 #1 Furn C#2 | 11 #2 Furn C#3 | 12 #3 Fun C#4 | 13 #4 Furn. C#5 | 14 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| hr:min | ft3 | | °C | °C | °C | psi | °C | °C | °C | °C | °C | °C | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| 12:00 | 3400.5 | Zero | 85 | 130 | 20 | 770 | 560/590 | 22 | 509 | 571 | 581 | 515 | |
| | Discharging $N_2$ | | | | | | | | | | | | |
| 12:10 | 3424 | 12.13 | 3430 | . At ~ | 12:20 | switch | to He | | | | | | |
| 12:26 | 3477 | 5.40 | 345 | 370 | 37 | 325 | 570/598 | 22.5 | 598/s | 594/s | 599/s | 598/s | 20/3.70 |
| | | Reduce flo | rate - | Attempting | to adjust | ~1 reflm | | | | | | | |
| 12:34 | 3521 | 3/.94 | → 3.2 | 35 | 27 → 35 | 29 | 2/3.1 → .165 | | | | | | |
| 12:42 | 3530 | .4 | 260 | 365 | 40 | 250 | 571/599 | 23 | 599/s | 594/s | 599/s | 595/s | 4/3.60 |
| 12:47 | 3532 | 1.11 | Discovered | that one $H_2$ | bottle | has only | 1750 | psi pressure, | | | | | |
| 1:15 | 3571 | 1.423 | 220 | 365 | 45 | 175 | -11- | 20 | -11- | -11- | -111 | -11- | 42/29.5 |
| 1:25 | 3581 | Conversion to | $H_2$ starts | | | | | | | | | | |
| 1:43 | | bottle $H_2$, | 3600 psi, | system is | being | pressurized, | no flows, | heaters | off | | | | |
| 1:45 | | | | | 1000 | 388/446 | 38 | 462 | 498 | 517 | 504 | | |
| 1:48 | | | | | 1300 | | | | | | | | |
| 1:49 | | | | | 1400 | 362/290 | 35 | 442 | 488 | 512 | 502 | | |
| 1:50 | | | | | 1630 | 346/374 | 35 | 424 | 478 | 508 | 498 | | |
| 1:53 | 3596 | | Bottle | 1800 | Pumping | to | 5200+26 | psi bottle | | | | | |
| 1:55 | | | 200 | 330 | 50 | 1610 | 341/369 | 29 | 394 | 456 | 491 | 474 | |
| 2:00 | | | 3850 | psi equalized | pressure | | Stripwater | steam | temp | on, | flows on | | |
| | Flow | is extremely | slow | | | | | | | | | | |
| 2:04 | 3610 | No | flow | | | | | | | | | | |
| 2:08 | Heaters | off | 2:10 | Pressure | 3500 | | | | | | | | |
| 2:14 | Dim Furnace | on | Vlc | wrong to | control | out | 2:17 | Anil's | action | -Lights | | | |
| 2:18 | 3615 | | 3180/2050 | bottle | | | | | | | | | |

B-7

TEST # 5 FEBRUARY 5, 1983 TABLE 5



| 1 Time | 2 Totaliz. | 3 Flow-rate | 4 Reactor inlet | 5 Zone #4 | 6 Reactor exit | 7 Reactor press. | 8 Pre-heat F#1 | 9 Coal F. C#1 | 10 #1 Furn C#2 | 11 #2 Furn C#3 | 12 #3 Furn C#4 | 13 #4 Furn. C#5 | 14 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| hr:min | ft3 | | °C | °C | °C | psi | °C | °C | °C | °C | °C | °C | |
| 2:22 | $H_2$ bottles are being shut off | | | | on carrier | | | | | | | | |
| 2:27 | 3610+ | 29 | 00psi | 1200 | | | Vent line | | | | | | |
| 2:35 | 3610 | -11- | | $H_2$ in being shut | | | off | 2:37 | He in being | | connected | 40 psi | |
| 2:43 | 3626 | 10÷ | 1.97 = 5.08 | | | | | | | | | | |
| 2:49 | 3650 | | Bottle | pressure to 1400, He | | | | | | | | | |
| 2:52 | 3654 | 6÷1.73 = 3.47 | | Bottle 132 | | | | | | | | | |
| 2:53 | 3662 | | | | | | | | | | | | |
| 2:59 | 3668 | $H_2$ in | being | connected | | 2325psi / 2100 | | 3:01 Set | | 2000 psi reached | | | |
| 3:02 | 3668.5 | No flow 2650 | | | | | | | | | | | |
| 3:06 | -11- | Start | venting | to | reduce | line | pressure to | | | | | | |
| 3:10 | | Bottle | 2600 | line | 1500 | | | | | | | | |
| 3:17 | -11- | | | | | | | | | | | | |
| 3:18 | 3672.5 | 3/2.3 = 1.3 | | | | 1500 | | | | | | | |
| 3:24 | 3686 | 9÷1.6 = 5.6 scfm | | | | 1000 | Bleed | this | change | to | He | | |
| 3:38 | 37156 | | | | | | | | | | | | |

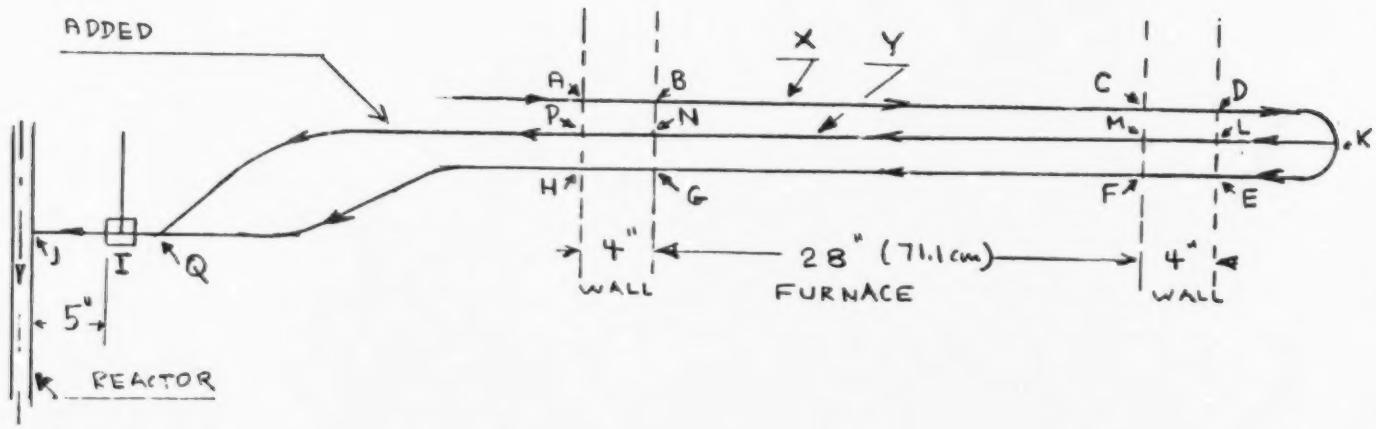



Figure 1

PRE-HEATER DIMENSIONS (Scale Approx 1:10)

1. 1/4" Schedule 40 pipe (0.540" O.D., 0.364" ID., 0.088" WALL)
   Internal cross section 0.671 $cm^2$. Internal surface 2.9 $cm^2/cm$ External surface 4.31 $cm^2/cm$

| | Definition | Kilborn | | Kilborn/ORF | |
|---|---|---|---|---|---|
| | | Reference | Length, cm | Reference | Length, cm |
| 1 | Within the Furnace | BC, FG | 142.4 | BC,FG,MN | 213.4 |
| 2 | Within the Walls | AB,CD,EF,GH | 40.6 | AB,CD,EF,GH,LM,NP | 61.0 |
| 3 | Outside, med. $H_2$ temp. | DE | 39.4 | DE,KL | 57.1 |
| 4 | Outside, high $H_2$ temp. | HJ | 83.3 | HJ,PQ | 136.7 |
| 5 | Total | | 305.6 | | 468.1 |

# APPENDIX C

## PROCEDURES AND RESULTS OF EFFLUENT (PRODUCT) GAS ANALYSIS

RESEARCH
FOUNDATION

SHERIDAN PARK RESEARCH COMMUNITY
MISSISSAUGA ONTARIO CANADA L5K 1B3 • (416) 822 4111 • TELEX 06 982311

Mr. A. J. Last
Engineering Services

January 6, 1984.

**REPORT NUMBER** 47-40007-84-001

**IDENTIFICATION** Your W.O. #14257

**SPECIFICATIONS OF ORDER** Determine levels of Carbon Monoxide (CO), Methane ($CH_4$), Carbon Dioxide ($CO_2$), Ethylene ($C_2H_4$) and Ethane ($C_2H_6$) in the supplied gas samples.

## PROCEDURE

The samples were delivered in pressurized gas jars. The jars were connected to the gas sampling valve (3 mL loop) of the GC by means of a transfer line fitted with a needle valve. The GSV was flushed with the sample at ~ 60 mL/min for 30 sec and the valve position switched to inject. Each sample was injected three times and the average of the peak areas taken for quantitation.

Quantitation was achieved by injecting a gaseous mixture of known concentration, of the compounds of interest. A comparison of the peak areas to the standard concentrations in mole per cent was then made.

Peak areas values were obtained by digital integration of the electrometer output. This was achieved by use of a Spectra Physics SP-4020 data interface and a SP-4060 remote terminal connected to a SP-4000 central processor.

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GC Parameters

| | |
|---|---|
| Instrument | : Varian 1400 with Thermal Conductivity Detection |
| Column | : 4' x 1/8" Carbosieve B 60/80 |
| Column Temperature | : Isothermal (35°C) for 2 min and then temperature programmed @ 20°C/min to 175°C |
| Carrier Gas | : Helium |
| Cell Voltage | : 200 Ma |
| Injector Temperature | : 200°C |
| Detector Temperature | : 200°C |

Typical GC/TCD profiles are shown in Figures 1 and 2.

RESULTS

The results of the sample analyses are shown in Table 1.

The November 28th samples showed only a large peak at a similar retention time to Nitrogen. However, these jars were at atmospheric pressure, so sufficient sample gas may not have been collected to enable us to detect the specified compounds.

J. Brady
Senior Technologist
Environmental and Chemical
Engineering Division

JB/bmc
Attachments

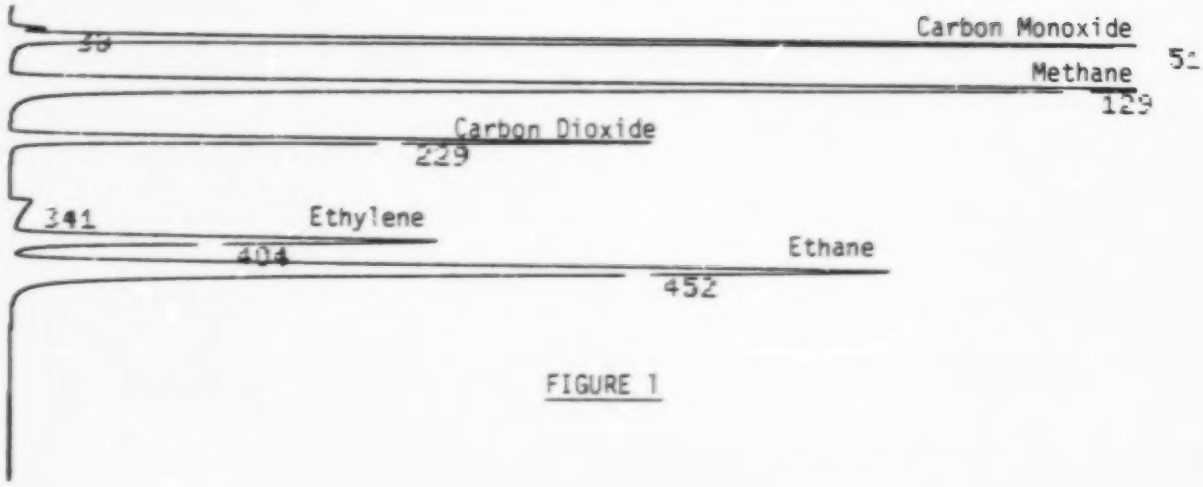


FIGURE 1

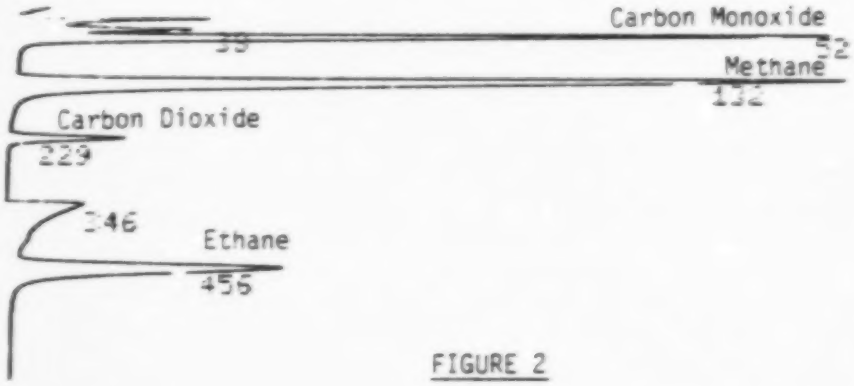


FIGURE 2

TABLE 1

| Client Identification | | Mole Per Cent | | | | |
|---|---|---|---|---|---|---|
| | | CO | $CH_4$ | $CO_2$ | $C_2H_4$ | $C_2H_6$ |
| Nov. 28 | | | | | | |
| 3 - 20 | | < DL* | < DL | < DL | < DL | < DL |
| 1 - 12 | | < DL | < DL | < DL | < DL | < DL |
| 2 - 20 | | < DL | < DL | < DL | < DL | < DL |
| Nov. 29 | | | | | | |
| Test III | 1 | 0.21 | 0.17 | < DL | < DL | < DL |
| | 2 | 0.26 | 0.75 | 0.02 | 0.01 | 0.08 |
| Dec. 9 | | | | | | |
| Run 8 | 1 | 0.3 | 0.63 | 0.24 | 0.03 | 0.3 |
| | 2 | 0.3 | 0.71 | 0.03 | - | 0.13 |
| Dec. 14 | | | | | | |
| Run 9 | 1 | 0.35 | 0.97 | 0.016 | - | 0.15 |
| | 2 | 0.33 | 0.80 | 0.084 | Trace** | 0.42 |
| | 3 | 0.32 | 0.76 | 0.18 | Trace | 0.36 |
| Dec. 15 | | | | | | |
| Run 10 | 1 | 0.33 | 1.09 | < DL | < DL | 0.112 |
| | 2 | 0.35 | 1.29 | < DL | < DL | 0.22 |
| | 3 | 0.40 | 1.53 | < DL | < DL | 0.24 |
| | 4 | 0.40 | 1.68 | < DL | < DL | 0.26 |
| Dec. 16 | | | | | | |
| Run 11 | 1 | 0.16 | 0.38 | Trace | < DL | 0.03 |
| | 2 | 0.26 | 0.55 | 0.03 | < DL | 0.16 |
| | 3 | 0.37 | 0.74 | 0.03 | < DL | 0.18 |
| * Detection Limit | | 0.004 | 0.009 | 0.004 | 0.006 | 0.007 |

** Trace: indicates presence of peak but below measurable levels.

MAPS NOT

FILMED

CARTES

OMISES

POOR

QUALITY

ORIGINAL